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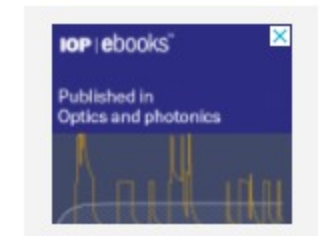
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Do it yourself: optical spectrometer for physics undergraduate instruction in nanomaterial characterization

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Do it yourself: optical spectrometer for physics undergraduate instruction in nanomaterial characterization

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Abstract

In this paper, we report on a homemade optical spectrometer using diffraction grating and image processing techniques. This device was designed to produce spectral images that could then be processed by measuring signal strength (pixel intensity) to obtain the light source, transmittance, and absorbance spectra of the liquid sample. The homemade optical spectrometer consisted of: (i) a white LED as a light source, (ii) a cuvette or sample holder, (iii) a slit, (iv) a diffraction grating, and (v) a CMOS camera (webcam). In this study, various concentrations of a carbon nanoparticle (CNP) colloid were used in the particle size sample test. Additionally, a commercial optical spectrometer and tunneling electron microscope (TEM) were used to characterize the optical properties and morphology of the CNPs, respectively. The data obtained using the homemade optical spectrometer, commercial optical spectrometer, and TEM showed similar results and trends. Lastly, the calculation and measurement of CNP size were performed using the effective mass approximation (EMA) and TEM. These data showed that the average nanoparticle sizes were approximately 2.4 nm and 2.5 ± 0.3 nm, respectively. This research provides new insights into the development of a portable, simple, and low-cost optical spectrometer that can be used in nanomaterial characterization for physics undergraduate instruction.

Keywords: optical spectroscopy, do it yourself (DIY), nanomaterial characterization, carbon nanoparticles

(Some figures may appear in colour only in the online journal)

1. Introduction

The development of a low-cost optical spectrometer to characterize the absorbance spectra of nanomaterials is important for potential use in undergraduate instruction [1, 2]. Many technological approaches using various schematic and detecting principles have been proposed. Currently, commercial optical spectrometers employ the diffraction pattern principle [3]. Optical spectrometers (especially UV–visible spectrometers) have been used in many applications, such as investigating semiconductor energy gaps, measuring particle sizes (or thin film thicknesses), and determining the concentration of solutions in industry, research institutions, or educational laboratories [4].

A low-cost, portable optical spectrometer to observe electromagnetic spectra using a diffraction grating has been developed by other researchers and educators [5, 6]. However, these devices have various weaknesses, including being difficult to set up, requiring a fairly large physical area, and being difficult to position properly. Therefore, it is necessary to develop new optical spectrometer devices to overcome these weaknesses [7]. A diffraction grating-based optical spectrometer combined with digital image processing provides a potential schematic that will likely be smaller in size, simpler to use for taking real-time measurements, cheaper to fabricate, and lower in energy usage. In addition, this optical spectrometer is projected to be accessible for use in student laboratory instruction, especially for the purpose of nanoparticle characterization.

Carbon nanoparticles (CNPs) are a type of semiconductor nanoparticle that have several unique characteristics, such as relatively uniform size, dispersibility in water, superior optical properties, and a facile synthesis method [8–10]. Moreover, accurate measurement of CNP concentration and particle size has an important role in the development of carbon applications in low-cost research in educational laboratories. Therefore, it is essential to develop comprehensive instructions to construct a measurement method that is simple, inexpensive, and nondestructive for analyzing concentration and particle size.

Currently, there are no complete studies regarding the characterization of CNP concentration and particle size using an optical spectrometer. Therefore, this study aimed to develop a diffraction grating-based optical spectrometer that uses image processing. This device was used to measure the particle size and concentration of CNPs. This research provides new insights into developing a portable, simple, and low-cost optical spectrometer that can be used in nanomaterial characterization.

2. Experimental overview

An optical spectrometer works by comparing the signal strength of the transmitted electromagnetic light coming out of the sample and the incident light entering the sample. The electromagnetic spectrum is obtained when the light passes through a diffraction grating and forms a diffraction pattern because the pattern formed depends on the wavelength of the light. Thus, the signal strength ratio of the transmission and incident light reveals the transmittance or absorbance spectra of the sample. Figure 1 shows a schematic diagram and prototype of the optical spectrometer designed in this study. Simply, this device consisted of: (i) a light source, (ii) cuvettes or a sample holder, (iii) a slit, (iv) a diffraction grating, and (v) a CMOS camera (webcam). A white-light-emitting diode (w-LED) was used as the light source to produce a wavelength spectrum between 400 and 720 nm. Then, the sample holder was set to hold a

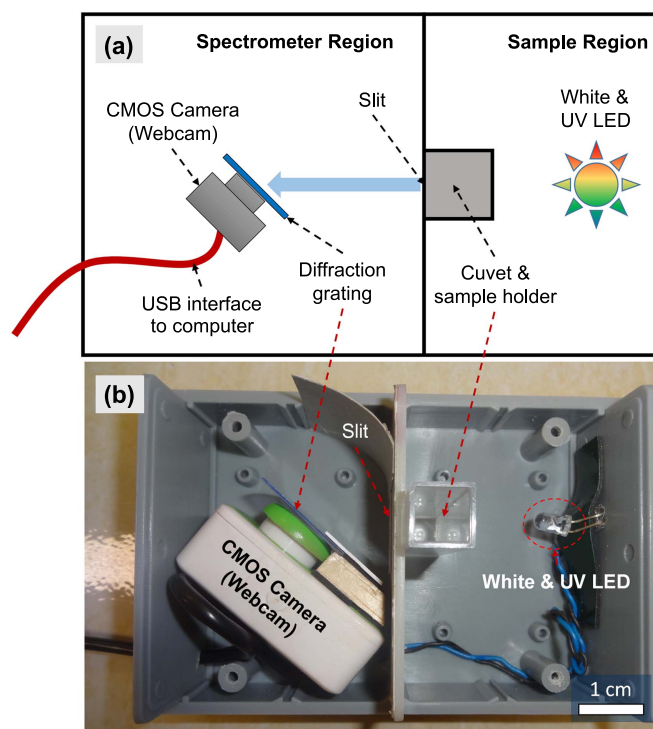


Figure 1. (a) Schematic diagram and (b) prototype of the diffraction grating and image processing-based optical spectrometer.

plastic cuvette with dimensions of $1 \times 1 \text{ cm}^2$. A digital video disk (DVD) was used as the diffraction grating. To create a diffraction grating from a standard 4.7 GB DVD do the following: (a) split the two layers of the disk using a knife along the edge; (b) cut a suitable piece of the transparent side to make the transmission grating. The grating so obtained has a groove spacing of 740 nm (13 500 lines/mm). To achieve the best results, installation of the diffraction grating must be based on the characteristics of the selected diffraction grating. Therefore, the diffraction surface orientation was set at 40° – 45° from the direction of light propagation. Meanwhile, the CMOS camera (webcam) was used to capture the digital image of the rainbow-like diffraction pattern. Furthermore, the digital images were processed to produce the transmittance and absorbance spectra of the sample.

The process of determining the size of the nanoparticles using a diffraction grating and image processing-based optical spectrometer is shown in figure 2. The CNPs were synthesized using citric acid and urea (as a carbon and fuel source, respectively) held at 190°C for 2 h to obtain black powder carbon [10]. The solution of CNPs was prepared by dispersing this carbon powder in water at several carbon concentrations (g l^{-1}) using the dilution method. Several steps are required to measure the size of nanoparticles using a diffraction-grating-digital-image-processing-based optical spectrometer, including (a) preparing the solution, (b) capturing the digital image of the diffraction pattern of the incident (background) and transmitted light of the samples, and (c) processing the image of the diffraction pattern. The image processing comprises several steps, including (a) converting

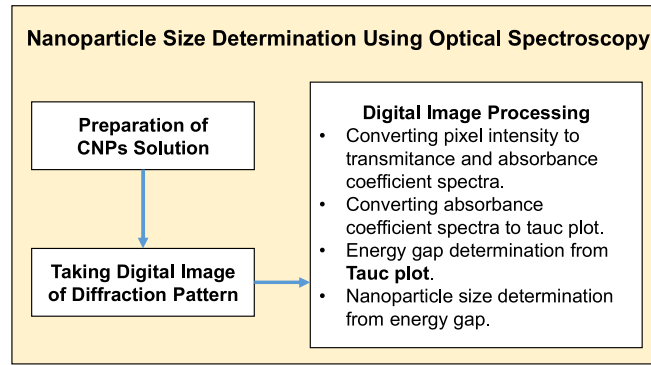


Figure 2. Flow chart describing the process of determining the size of the nanoparticles using a diffraction grating and image processing-based optical spectrometer.

the digital image into the transmittance spectra by measuring the signal strength (usually referred to as the pixel intensity) of the diffraction pattern, (b) converting the transmittance spectra into the absorbance coefficient spectra and Tauc plot, (c) calculating the optical band gap energy of the CNPs from the Tauc plot, and (d) calculating the CNP sizes from the optical band gap. Several powerful open source software packages can be used to easily process and manipulate the digital images (RGB intensity) into signal strength data with high-quality results, such as Tracker 4.94, which is a free Java video analysis tool developed by the Open Source Physics Project (<http://physlets.org/tracker/>) or Cell Phone Spectrometer (CPS) by Alexander Scheeline and Kathleen Kelley (<http://scheeline.scs.illinois.edu/~asweb/CPS/>) [11, 12]. In the analysis of the video images in this study, the line profile tool of Tracker 4.94 software was used. Additionally, optical and morphological characterization were performed using a commercial optical spectrometer (LD Didactic SpectraLab) and tunneling electron microscope (TEM, JEM-3000F, JEOL, Tokyo, Japan) to confirm and support the quality of the spectral data and morphology, as well as the calculation results from this optical spectrometer.

3. Results and discussion

3.1. Optical spectra of the CNP solution

The digital image processing was conducted by analyzing the signal strength of the spectral image from the blue $B(x_B, y_B)$ to the red $R(x_R, y_R)$ spectrum position across a straight line. The signal strength of the pixels at each pixel position $\lambda(x, y)$ in relation to the wavelength was calculated as follows:

$$I(x_B, y_B) = I(\lambda_{400\text{nm}}), I(x_R, y_R) = I(\lambda_{720\text{nm}}), \quad \text{and} \quad I(x, y) = I(\lambda)$$

with scalar ratio,

$$\frac{|B\lambda|}{|BR|} = \frac{\lambda - 400}{720 - 400} \quad (1)$$

$$\frac{|\lambda(x, y) - B(x_B, y_B)|}{|R(x_R, y_R) - B(x_B, y_B)|} = \frac{\lambda - 400}{720 - 400} \quad (2)$$

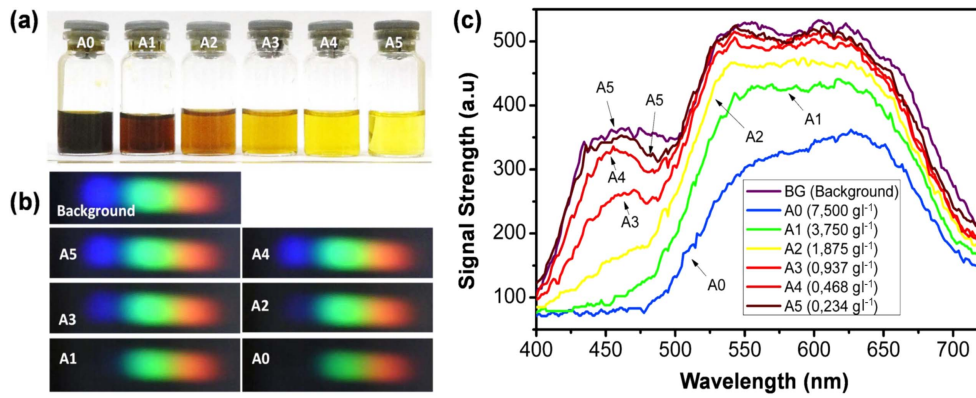


Figure 3. (a) Pictures of the prepared samples, (b) light diffraction pattern, and (c) optical spectra analysis for several concentrations of CNPs, 0.234 g l^{-1} to 7.5 g l^{-1} .

or

$$\frac{\sqrt{(x - x_B)^2 + (y - y_B)^2}}{\sqrt{(x_R - x_B)^2 + (y_R - y_B)^2}} = \frac{\lambda - 400}{720 - 400} \quad (3)$$

and then,

$$I(\lambda) = I \left(400 + (720 - 400) \left(\frac{\sqrt{(x - x_B)^2 + (y - y_B)^2}}{\sqrt{(x_R - x_B)^2 + (y_R - y_B)^2}} \right) \right) \quad (4)$$

where the analysis of signal strength was conducted for all transmitted and incident light diffraction patterns (background intensity).

Figure 3(a) shows the prepared CNP solutions with various concentrations. As shown, the prepared samples had different color densities depending on the carbon concentrations, where a darker color indicates a higher concentration. Figures 3(b)–(c) show the digital images of the diffraction pattern and optical spectra of the CNP solutions for various concentrations. The optical spectra were obtained by image processing the diffraction patterns by calculating the signal strength using equation (4). These results prove that the concentration of CNPs affected the signal strength of the diffraction pattern spectrum. Analysis of the diffraction pattern image and optical spectra indicated that there was significant energy absorption by the CNP solutions in the blue color region.

3.2. Transmittance and absorbance spectra of CNP solutions

Based on the optical spectra shown in figure 3(c), the transmittance of the CNP solution was calculated using equation (5):

$$T(\lambda) = \frac{I(\lambda)}{I_o(\lambda)} \quad \text{or} \quad \%T = T \times 100\% \quad (5)$$

where $I(\lambda)$ is the transmitted, $I_o(\lambda)$ is the incident signal strength, and $T(\lambda)$ is the transmittance spectra.

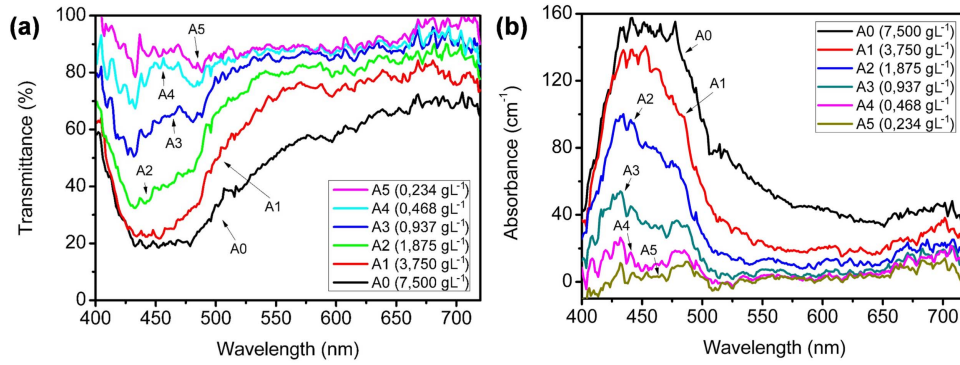


Figure 4. (a) Transmittance and (b) absorbance coefficient spectra for various concentrations of CNPs from 0.234 g l^{-1} to 7.5 g l^{-1} .

The relationship between absorbance (A), transmittance (T), and concentration (C), known as the Lambert–Beer law, which is expressed in equation (6):

$$A = -\log\left(\frac{I(\lambda)}{I_0(\lambda)}\right) = -\log T = \varepsilon dC \quad \text{or} \quad \alpha = \frac{A}{d} = -\frac{1}{d} \ln(T) = \varepsilon C \quad (6)$$

where α is the absorbance coefficient (m^{-1}), ε is the molar absorbance value ($\text{mol}^{-1} \text{ m}^2$), C is the concentration of particles in the solution (mol m^{-3} or g l^{-1}), and d is the path length (m) [3].

The transmittance and absorbance spectra calculation results for the solutions with different concentrations of CNPs are shown in figure 4. These results prove that the transmittance ratio and absorbance value depend on the concentration of CNPs. As shown in figure 4, the solution of CNPs had an absorption spectra in the visible region (400–720 nm), with the highest absorbance occurring in the blue–yellow spectrum regions (400–500 nm, or 2–3 eV). This result shows that the CNPs had a wide optical band gap just like semiconductor materials. As we know, carbon bulk has a very small optical band gap ($E_g \cong 0.05$) that enlarges when it turns into CNPs [13]. Thus, there is a correlation between the size of the particles and the optical band gap of the CNPs. Some of the literature on nanomaterials explains that the quantum confinement effect caused by changes in particle size will affect the energy band gap of a nanomaterial [14]. Therefore, the optical band gap of carbon particles could be used to measure the size of the CNP quantitatively. In addition, it is interesting to see the linear relationship between the molar absorbance value and the concentration of CNPs.

As shown in figure 5, we compared the transmittance spectra obtained from our proposed optical spectrometer (UIN MOS) with that obtained from a commercial spectrometer (LD MOS). The comparison results show that the transmittance spectra of both our apparatus and the commercial optical spectrometer have similar transmittance ratio and absorbance peak characteristics. In addition, a comparison of the measurements indicated that the transmittance ratio and absorbance value were proportional to the concentration of CNPs. Thus, we concluded that the quality of our proposed optical spectrometer is almost comparable to a commercial spectrometer.

3.3. Concentration analysis of CNP solutions

The concentration of the CNP solution is related to the experimentally observed absorbance value (A) as shown in equation (6), via the molar absorbance value (ε) and the path length of

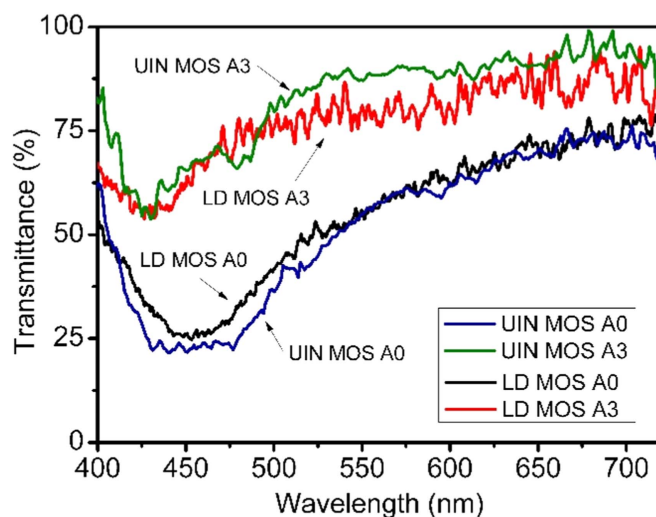


Figure 5. Data comparison between the transmittance spectra obtained from our proposed optical spectrometer and that obtained from a commercial spectrometer (LD Didactic SpectraLab).

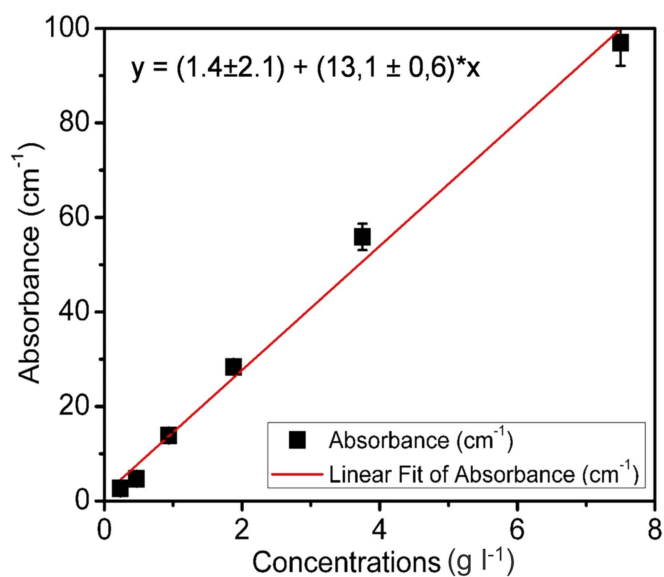


Figure 6. Relationship between the CNP concentration and absorbance at 500 nm with 5% error bars.

the spectrometer (d), which is usually 1 cm. Figure 6 shows the relationship between the concentration of CNPs and the absorbance value at 500 nm. As shown, there was a linear relationship between the CNP concentration and the absorbance value. The slope of $13.1 \pm 0.6 \text{ mol}^{-1} \text{ m}^2$ is the molar absorbance value (ϵ) of the CNPs at 500 nm.

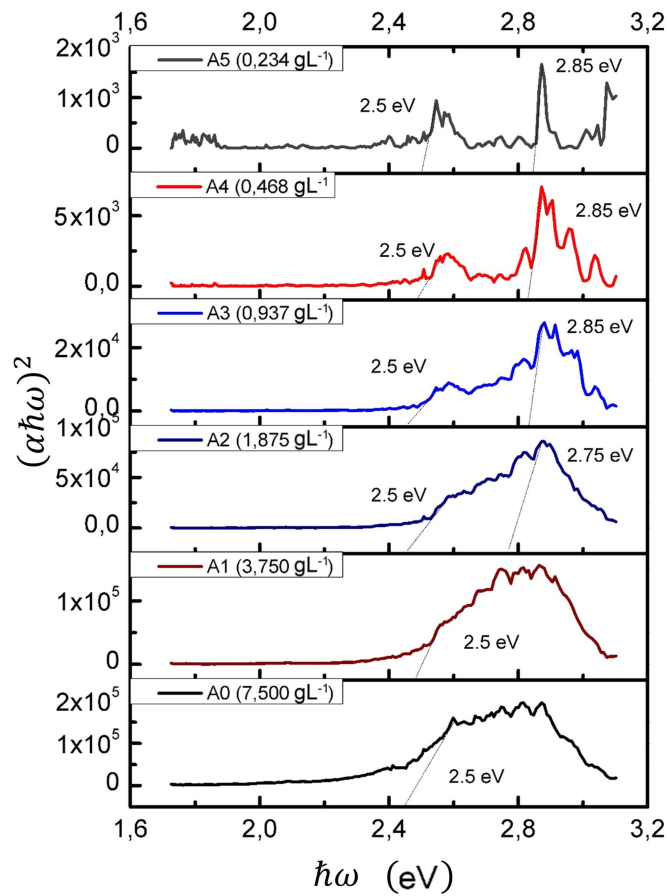


Figure 7. Tauc plot from the absorbance spectra for several CNP concentrations, from 0.234 g l^{-1} to 7.5 g l^{-1} .

3.4. Band gap and nanoparticle size analysis

The optical band gap can be calculated using the relationship determined by the Tauc plots:

$$(\alpha\hbar\omega) = C(\hbar\omega - E_g)^n \quad (7)$$

where C is a constant, α is the absorbance coefficient, E_g is the optical band gap of the material, and n depends on the type of transition ($n = 1/2$ and 2 for a direct band gap and indirect band gap, respectively) [15, 16]. The optical band gap was determined based on the point of intersection on the x -axis of a straight line drawn from the linear portion with the largest slope on the Tauc plots indicating conformity with equation (7) under ideal conditions. The calculated optical band gap from the Tauc plots, considering that it was a direct band gap, was found to be 2.5–2.85 eV for the CNPs, as shown in figure 7. The calculation results showed that all samples had a wide optical band gap value, and this corresponds to the characteristics of semiconductor nanomaterials. Furthermore, figure 7 shows that the DIY optical spectrometer has excellent accuracy for high CNP concentration, but low level accuracy for low CNP concentration. This low level accuracy could be caused by low quality of webcam that used in this experiment. Thereby, the CNPs were monodisperse and not

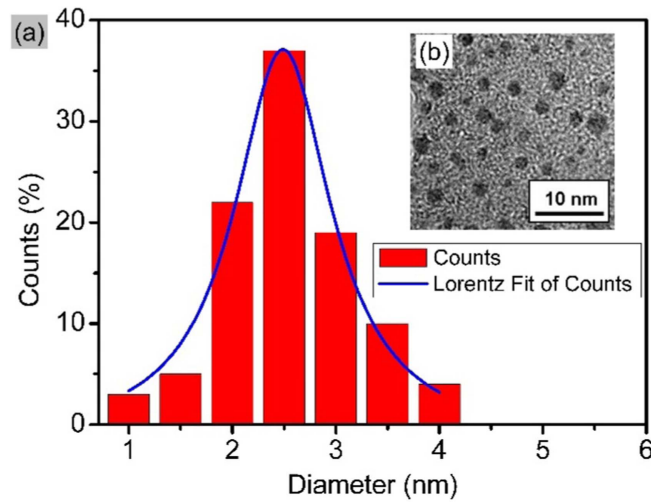


Figure 8. (a) Particle size distributions and (b) TEM image of CNPs with average size of 2.5 ± 0.3 nm.

agglomerated, even at high concentrations, due to the dominant presence of a functional bonding surface (OH⁻) so the carbon particles could easily interact with polar solvents like water. Another explanation for the lack of agglomeration is that the carbon particles were neutral and/or negatively charged, which led to a repulsive Coulomb force between the CNPs so they could not easily agglomerate.

It is already known that the size of particles affects the optical band gap of a nanomaterial. The relationship between the size of the nanoparticles and the optical band gap is explained by the Brus law equation (EMA, effective mass approximation), as follows [15, 17]:

$$E_g^* = E_{g0} + E_{n,l}^{\text{conf}} = E_{g0} + \frac{\hbar^2 \pi^2}{2R^2} \left(\frac{1}{m_e} + \frac{1}{m_h} \right) + \text{small terms.} \quad (8)$$

In addition, we can change the Brus law equation so that energy is expressed in eV, the diameter in nanometers, as well as the electrons mass (m_e) and holes mass (m_h) in the free electron mass (m_0), as follows [17]:

$$E_g^* = E_{g0} + \frac{14.84}{R^2} \left(\frac{1}{m_e} + \frac{1}{m_h} \right) + \text{small terms} \quad (8a)$$

by assuming the prepared CNPs are in the carbon graphitic phase with a bulk energy gap (E_{g0}) equal to zero, the effective electron mass is $0.19 m_0$, and the effective hole mass is $0.25 m_0$ [9]. Therefore, according to equation (8), a CNP with an optical energy gap at 2.5–2.85 eV has a particle size of approximately 2.4–2.7 nm, respectively.

Additionally, TEM was used to determine the average size of the CNPs. A TEM image of the CNPs was characterized to verify the particle size distribution and the presence of monodisperse CNPs, as shown in figure 8. A histogram of the particle size distribution and standard deviation were created and calculated, respectively. From the analysis of the TEM image, it was found that that the average size of the CNPs was 2.5 ± 0.3 nm. The TEM result is similar to the calculated particle size of the prepared sample (~ 2.4 – 2.7 nm) using the optical spectra.

4. Summary

A diffraction grating and image processing-based optical spectrometer for use in CNP size and concentration measurements has been developed. When comparing the data obtained from our proposed optical spectrometer with that obtained from a commercial optical spectrometer, we found similar results and trends. In addition, the results from the calculation and measurement of CNP size using EMA and a TEM showed that the average size of the CNPs was 2.4 nm and 2.5 ± 0.3 nm, respectively. This study proves that a diffraction grating and image processing-based optical spectrometer can provide powerful results, with a simple, low-cost apparatus that is able to determine nanoparticles sizes and measure concentrations. Therefore, this spectrometer device has the potential to be further researched and developed so it may become more accessible for use in student projects or low-cost research or educational laboratories.

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