Laser ablated titanium oxide nanoparticles in carbon quantum dots solution for detection of sugar using fluorescence spectroscopy

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Abstract

Titanium oxide nanoparticles were fabricated using the laser ablation technique in the carbon quantum dots aqueous solution. The ablation time and the particle size were in the range of 2 min to 10 min, and 21.46 nm to 16.07 nm, respectively. The fingerprint peaks and the particle shape in spherical form were obtained from Fourier transform infrared spectrum and transmission electron microscopy, respectively. The optical properties were tested using a UV–visible spectrometer, so the bandgap was obtained in the range of 3.44 eV to 3.7 eV. The prepared sample was used to detect the sugar and the sensitivity of the sample for the detection of glucose is greater than other sugar. The limit of detection is 0.01 ppm.

1. Introduction

Titanium oxide nanoparticles (TiO$_2$-NPs) are a family of metal oxide or ceramic. There are three types of TiO$_2$ in the crystalline form including, anatase, rutile, and brookite [1]. The TiO$_2$-NPs have promising properties including high chemical stability [2], wide bandgap [3], easy availability, biocompatibility, non-toxicity, photocatalyst [4], and cheap price. The TiO$_2$-NPs can be used in organic solar cells [5], dye synthesis solar cells [6], sensors [7], biosensors, photo-electrochromic components food, and cosmetics [8]. The TiO$_2$-NPs were prepared using deposition methods [9], spray pyrolysis [9], sonochemical and microwave-assisted methods [9, 10], hydro/solvothermal methods [9, 11], the metal–organic chemical vapor deposition (MOCVD) [12], and electrochemical methods [9]. Nath et al [13] prepared TiO$_2$-NPs in an aqueous solution and reported that the nanoparticles were formed in a spherical shape, and the particle size was about 45 nm [13]. The effect of ambient pressure on the crystallization of TiO$_2$-NPs was investigated by Matsubara et al [14] who found that the particle size depends on ambient pressure [14].

Laser ablation is a process and dynamic method to prepare the nanomaterial. A high-power laser beam uses to realize material such as metal or ceramic from the surface of the target. Laser ablation is considered and applied for the production of metal nanoparticles, deposition of thin metallic and dielectric films [15].

Laser ablation was considered to prepare the gold nanoparticles [16–18], silver nanoparticles [17, 19, 20], ZnO nanoparticles [21–23], and titanium oxide nanoparticles [24, 25]. The pulsed laser was used to ablate the titanium target so that the TiO$_2$-NPs can be fabricated in an aqueous solution [26]. The TiO$_2$-NPs also formed in spherical shape in the presence of a surfactant sodium dodecyl sulfate (SDS) solution, and the particle size was about 40 nm. Giorgetti et al [27] fabricated the TiO$_2$-NPs in the distilled deionized water using pulsed Q-switch Nd:YAG laser and they investigated the effect of pressure and temperature on the crystalline of TiO$_2$-NPs. Consequently, the laser ablation technique can be used to prepare the TiO$_2$-NPs, and this technique is a green and simple method of synthesizing the TiO$_2$-NPs from a titanium target.
Carbon quantum dots (CQD) and graphene quantum dots (GQD) belong to the class of nanomaterial with a 0-dimension size [28]. CQD has unique properties including, the particle size of fewer than 10 nanometers, high fluorescence intensity, high-temperature stability, biocompatibility, low toxicity, resistance to photobleaching [29, 30], and solubility in water and organic solvents. Hence, the synthesis, properties, and application of CQD and GQD are the subjects of interest in nanoscience, nanobiotechnology, nanoelectronics, nanophotonics [28], and optoelectronic device [31]. Organic Quantum dots [32, 33], CQD, and GQD have the potential to prepare and improve optoelectronic devices such as solar cells and light emitted diodes.

The composition of titanium oxide and carbon quantum dots can enhance the photocatalyst [34], antibacterial [35], and fluorescence properties of CQD. Hence, it is important to pay attention to the properties of TiO2-NPs/CQD composite.

Sugar is an important component in food, beverage, and human blood. The main sugar that can be found in food and beverage are glucose, sucrose, and fructose. Therefore, many methods have been considered for measuring the level of glucose, fructose, and sucrose. The spectroscopy methods, that are important to investigate the sugar, are Raman spectroscopy [35], colorimetric technique [36], fluorescence spectroscopy [37], and electrochemistry methods [38]. Moreover, numerous physical sensors were used to detect the sugar including the surface plasmon resonance sensor [39], ultrasonic sensor [40], fiber optics sensor [41], photoacoustic sensor [42], microwave [43] resistance sensor [41], and capacitor sensor [42]. Therefore, the detection and measurement of low concentrations of glucose, fructose, and sucrose are significant topics in chemistry, applied physics, electronics, and medicine.

In the last decade, nanomaterials were considered to detect sugar [37]. The significant nanoparticles that used to sense the sugar are carbon nanotube [43], graphene [44], gold nanoparticle [45], barium ferrite nanoparticles [46], magnetite nanoparticles [47], zinc oxide nanoparticles [48, 49], and titanium oxide nanoparticles [50]. Titanium oxide nanoparticles were used to detect the sugar using the electrochemistry technique [51], and the plasmonic method [52]. Consequently, the titanium oxide nanoparticles have the potential to detect sugar.

In this study, the TiO2-NPs were fabricated in the CQD solution from biochar using high power pulsed laser. The prepared samples were characterized using Fourier transform infrared spectroscopy, transmission electron microscopy, UV-visible spectroscopy, and fluorescence spectroscopy. The prepared sample was used to detect glucose, fructose, and sucrose using fluorescence spectroscopy.

2. Materials and methods

2.1. Regents
Biochar from an empty fruit bunch (EFBB), isopropanol (assay 98%), glucose, fructose, sucrose and high purity Titanium plate were provided by the Parka Go Green Sen Bud, Merck Company (Darmstadt, Germany), and Sigma Aldrich, respectively. Solvents used were an analytical grade.

2.2. Preparation of carbon quantum Dot
The preparation of CQD was reported in [53, 54]. Briefly, the CQD was prepared from empty fruit bunch biochar (EFBB) in acid-free synthesis. The EFBB was selected as the carbon source, while isopropanol was used as a co-solvent in a 3:1 ratio water/isopropanol mixture. In a typical process, 0.06 g of EFB biochar was added to a steel tube reactor. Then, 6 ml of a co-solvent mixture was added to the same tube and then sonicated for 5 min before being placed into an oven at a temperature of 250 °C for 60 min. Upon completion of the heating process, the steel tube was left immersed in a water bath to cool for about 4 h. The black liquid that was produced was purified using centrifugation (4000 rpm, 5 min) to eliminate the residual biochar [55–58]. The supernatant containing CQD was then withdrawn and kept for further characterization.

2.3. Laser ablation setup
The TiO2-NPs were prepared using laser ablation. The setup (figure 1) contains Nd:YAG (1064 nm), stirrer, lens, sample tank, and Ti-plate (purity 99%). The repetition rate was 40 Hz and the Ti plate was the irradiated duration of 2, 6, and 10 min (ablation time). The Ti plate was immersed in 40 ppm concentration of CQD solution and the stirrer dispersed the nanoparticles in the solution. The prepared samples were characterized using UV-visible spectroscopy (UV-vis), transmission electron microscopy (TEM), and Fourier transforms spectroscopy (FT-IR) [53, 59, 60].
3. Results

Figures 2(a) and (b) show the FT-IR spectra of pure CQD and TiO$_2$-NPs/CQD solution at 10 min ablation time which is used for characterizing the nanofluid samples to prove the formation of TiO$_2$-NPs in CQD solution. The main peaks appeared at 3253.85, 3263.03, 1635.49, 1620.57, 1468.24, 1388.24, 1378.82, 1126.22 and 1126.51 cm$^{-1}$. The peak at 3253.85 and 3263.03 cm$^{-1}$ are related to the stretching vibrations of the $\text{–OH}$ groups\cite{54, 61}, and the peaks at 1620.57 and 1635.49 cm$^{-1}$ are related to the stretching vibrations of the carboxyl groups. The peaks at 1388.24, 1378.82 cm$^{-1}$ assigned the C=C\cite{54, 61} stretching vibration on the benzyl ring of CQD. These peaks appeared at 1126.51 and 1126.22 cm$^{-1}$ corresponded to the asymmetric and symmetric stretching vibrations of the C–O–C groups\cite{62–64}. The peaks at 1468.24 cm$^{-1}$ assigned the stretching vibration of Ti–O–Ti and it is related to TiO$_2$-NPs\cite{65} which confirmed the TiO$_2$-NPs formed in the CQD and the functional group at the edge of the molecule capped it. Figures 3(a) and (b) show the XRD patterns for the pure CQD and TiO$_2$-NPs/CQD composite, respectively. The main peaks appeared at 21.5°, 21.6°, 38.2°, 47.5°, 54.2°, and 62.4°. The broad peak at 21.5° and 21.6° is related to the crystalline lattice plane (002) in CQD\cite{66}. Figure 3(b) shows the peaks at 38.2°, 47.5°, 54.2°, and 62.4° assigned the crystal plain at (101), (004), (200), (105),
and (204) of TiO$_2$-NPs [67], respectively. The XRD spectrum confirmed that the anatase TiO$_2$-NPs formed in the CQD solution [68].

Figure 4 shows the TEM images for TiO$_2$-NPs/CQD solutions. The images were analyzed and the average particle size was achieved. Consequently, the nanoparticles formed in the CQD solution in spherical form, with average particle sizes of 21.46, 19.96 nm, and 16.07 nm. The particle size depends on the ablation time and viscosity of the solution and the average size of TiO$_2$-NPs was reported in the literature is in the range of 2 to 60 nm [31, 69]. As a result, the particle size decreases with an increase in the ablation time. The anatase TiO$_2$-NPs were surrounded by CQD in the crystalline form [54]. Figure 4(a)(12) and (13) depict the CQD in the crystalline form and the particle size and lattice space are about 4 nm and 0.21 nm respectively.

Figures 5(a) and (b) depict the absorption and transmission of the UV-visible spectrum, respectively. The peak at 360 nm is related to CQD. The UV-visible spectrum intensity increased with an increasing the ablation time and the transmission decreased. Figures 5(a) and (b) show the absorbance and the transmissivity of the experiment results of UV-Visible spectra.

A relationship of transmissivity ($T$) and absorbance of the TiO$_2$-NPs/GQD composite for different samples can be expressed as follows [53, 70]:

\[
\text{absorbance} = \text{optical density} = -\log_{10} T
\]

The ratio of transmitted beam intensity to the light beam intensity ($I/I_0$) is the transmissivity. The optical density (OD) of the TiO$_2$-NPs/CQD composite is explained by the absorption coefficient ($\alpha$) and the optical path ($l$) as follows [71]:

\[
\text{OD} = 0.434 \times \alpha \times l
\]

Normally, the thickness of UV–vis cell is 10 mm and it is equal to the optical path. So, the absorption coefficients obtained in the 360 nm wavelength for pure CQD and 2, 6, and 10 min ablation times of TiO$_2$-NPs/CQD composite solution were 1.137, 1.548, 1.875, and 3.626, respectively. The imaginary part ($k$) of the refractive index for TiO$_2$-NPs/CQD has a relationship with the wavelength ($\lambda$) of a light beam and the absorption coefficient ($\alpha$) at this wavelength is as follows [70]:

\[
k = \frac{\alpha \times \lambda}{4\pi}
\]

Figures 6(a)–(c) show the variation of absorption coefficient, the imaginary part ($k$) of refractive index, and real part ($n$) of the refractive index for the TiO$_2$-NPs/CQD solution. They were obtained from the analysis of

Figure 3. The XRD patterns for (a) pure CQD and (b) TiO$_2$-NPs/CQD composite solution at 10 min ablation time.
UV–vis spectra from 330 nm to 800 nm. The refractive index of TiO$_2$-NPs/CQD solutions is a function of wavelength, and the imaginary part and the absorption coefficient increased when the ablation time increased.

The energy bandgap ($E_g$) of TiO$_2$-NPs/CQD was calculated from UV–vis spectra using the Tauc relation [72] containing the photon energy ($h\nu$) as follows:

$$[\alpha h\nu]^2 = K (h\nu - E_g)^n$$

where $\alpha$, $K$, and $n$ are the absorption coefficient and the constant, respectively, and $n$ is equal to 1 for the direct bandgap. Figure 6(a) shows the variation of absorption coefficient with wavelength and it is a function of light beam wavelength as follows:

$$\alpha(\lambda) = \frac{2.303}{t} A(\lambda)$$

Where $t$ and $A(\lambda)$ are the thickness of sample and absorbance, and $t$ is equal to 10 mm (1 cm). The optical band gap of TiO$_2$-NPs/CQD solution was obtained from the plot of $[\alpha h\nu]^2$ versus photon energy. Figure 6 shows the variation of the photon energy ($h\nu$) of the light beam for TiO$_2$-NPs/CQD solution in the different ablation times. Figures 7(a)–(d) show the optical band gaps results related to pure CQD, TiO$_2$/CQD in 2, 6, and 10 min. They were obtained from the extrapolation of the straight portion of the plot ($[\alpha h\nu]^2 = 0$) [73] and the values of the optical band gap ($E_g$) for pure CQD, TiO$_2$-NPs/CQD solution in 2 min, 6 min, and 10 min ablation times are 3.44 eV, 3.56 eV, 3.61 eV, and 3.70 eV, respectively. Figure 7(e) shows the variation of the optical band gap with ablation time. When the ablation time increased the distribution of TiO$_2$-NPs increased and the bandgap also increased. It shows that there is a strong interaction between CQD and TiO$_2$-NPs that requires more ablation time for inhomogeneous seeding of TiO$_2$-NPs on the surface of CQD. The value of abs, %T, absorption coefficient, and bandgap have been presented in table 1.
Figure 5. (a) Absorption and (b) Transmittance of UV–vis spectra for pure and TiO$_2$-NPs/CQD composite solution at 2, 6, and 10 min.

Figure 6. (a) Absorption coefficient, (b) imaginary part of refractive index, and (c) real part of the refractive index of pure CQD, TiO$_2$-NPs/CQD composite solutions at 2, 6, and 10 min.
Figure 7. Show the variation of $(\alpha h\nu)^2$ with $h\nu$ to calculate the bandgap for (a) pure CQD and TiO$_2$-NPs/CQD solution in (b), (c), 6, (d) 10 min ablation time, and (e) the variation of bandgap with ablation time.

Table 1. The value for $\alpha$, absorption coefficient, (n), real part, (k), imaginary part of refractive index, and bandgap at 360 nm for pure CQD and TiO$_2$-NPs/CQD composite that achieved from figures 5–7.

| Sample                  | $\alpha$ | n | k | Bandgap (eV) |
|-------------------------|----------|---|---|--------------|
| Pure CQD                | 0.45     |   |   | 3.44         |
| TiO$_2$-NPs/CQD 2 min   | 0.83     | 0.21 | 1.548 | 3.56 |
| TiO$_2$-NPs/CQD 6 min   | 1.32     | 0.08 | 1.875 | 3.61 |
| TiO$_2$-NPs/CQD 10 min  | 1.78     | 0.05 | 3.626 | 3.70 |

Figure 8(a) displays the fluorescence spectrum of pure CQD and TiO$_2$-NPs/CQD solutions at 2, 6, 10 min ablation times. The excitation and emission of CQD are at 258 nm and 335 nm, respectively; that is due to a series of emissive traps between $\pi$ and $\pi^*$ of C–C related to hydroxyl, carbonyl, and carboxyl as a functional group on CQD molecule. The fluorescence spectrum of TiO$_2$-NPs/CQD solutions shows the intensity peak of emission increased as increasing the ablation time (figure 8(b)) and decreasing the particle size of TiO$_2$-NPs because the cross-section increased. Consequently, TiO$_2$-NPs enhanced the fluorescence properties of CQD.

The maximum intensity peak in the fluorescence spectrum (figure 8(a)) corresponded to the TiO$_2$-NPs/CQD in 10 min ablation time with the maximum bandgap (3.70 eV). So, the TiO$_2$-NPs/CQD in 10 min ablation time was used to detect the sugar. Figure 9 shows the fluorescence spectrum for the detection of glucose, sucrose, and fructose. The different concentrations of sugar (0.01, 0.3, 2, 5, and 10 ppm) were mixed with the
TiO$_2$-NPs/CQD solution in 10 min ablation time. The excitation wavelength and the emission wavelength were at 258 nm and 335 nm, respectively. The peak intensity of TiO$_2$-NPs/CQD solution before mixing with sugar was used as a reference which was equal to $8.136 \times 10^4$ a.u. As a result, the intensity peaks decreased after interaction with sugar.

Figure 9(d) shows that when the concentration of glucose, sucrose, and fructose increased from 0.01 ppm to 10 ppm, the intensity peaks decreased linearly, and the slope of variation curve for glucose is greater than sucrose and fructose. It means that the sensitivity for the detection of glucose is greater than sucrose and fructose.

The fluorescence experiments were carried out repeatedly about 10 times to evaluate the stability of the fluorescence property of the TiO$_2$-NPs/CQD solution in 10 min ablation time. The average intensity of fluorescence peaks and the standard deviation for measurement of glucose, sucrose, and fructose were 47451 a.u., $2.11 \times 10^3$, 56028 a.u., $3.29 \times 10^3$, 67169 a.u., $3.77 \times 10^3$, respectively. The limit of detection (LOD) is proportional to the standard deviation and slope ($K$) of variation of fluorescence intensity with the concentration of sugar (glucose, sucrose, and fructose). The calculation was continued using figure 9(d), and the slope ($K$) of variation curve for glucose, sucrose, and fructose were obtained about 73.7784, 68.211, and 54.959 a.u./ppm, respectively. Consequently, the value of LOD for glucose, sucrose, and fructose was achieved about 8.573, 14.464, 20.584, respectively. It means that the minimum variation of fluorescence intensity for the detection of glucose, which can be detected, is smaller than sucrose and fructose.

The fluorescence mechanism for the detection of sugar is based on the transfer of electrons between TiO$_2$-NPs and CQD. CQD has a hydroxyl functional group at the edge of the surface and it can interact with sugar using van der Waals interaction (Figure 10).
The main mechanisms that could explain the fluorescence property of CQD and TiO$_2$-NPs/CQD for the detection of sugar are based on surface-related defective sites. It generally refers to any sites that have nonperfect sp$^2$ domains that will result in surface energy traps. Both sp$^2$ and sp$^3$ hybridized carbons, the carbonyl group as a localized electronic state, contribute to the CQD and TiO$_2$-NPs/CQD emissions in the blue range. This mechanism is like that the aromatic molecules are individually incorporated into solid hosts, thereby exhibiting CQD and TiO$_2$-NPs/CQD emissions due to the existence of multiple surface defects that have different excitation and emission properties [74,75]. As a result, the excitation of the molecule was about 258 nm and the emissions were at 335 nm and TiO$_2$-NPs enhanced the intensity of the fluorescence spectrum. The emission occurred from CQD and TiO$_2$-NPs provide the extra electron which contributes to enhancing the fluorescence and interaction with sugar.

4. Conclusions

The TiO$_2$-NPs/CQD composite aqueous solutions were prepared using the laser ablation technique. The ablation time was 2 min, 6 min, and 10 min. The analytical methods such as FT-IR, UV–vis, and TEM images were confirmed that the TiO$_2$-NPs formed in the CQD solution, and the nanoparticles formed in the spherical shape with particle sizes of about 21.46 nm, 19.96 nm, and 16.07 nm for ablation times at 2 min, 6 min, and 10 min, respectively, and TiO$_2$-NPs distributed in the CQD solution. The bandgap was calculated from UV–vis spectrum in the range of 3.44 eV to 3.70 eV. Therefore, when the ablation time increased from 2 min to 10 min, the particle size decreased, and the bandgap increased. The fluorescence spectrum showed that the intensity of the fluorescence peak increased when the ablation time or population of TiO$_2$-NPs increased. The
TiO2-NPs/CQD can be used to detect and recognize the sugar. As a result, the sensitivity to recognize the glucose was greater than sucrose and fructose. Consequently, the TiO2-NPs formed in the CQD aqueous solution using the laser ablation method, and it was observed that the particle size can be controlled by time and it has the potential to sense the sugar.

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