

Oxygen free graphene/TiO₂ nanocomposite synthesis technique for dye-sensitized solar cells photo-anode

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ABSTRACT

This article presents the techniques for the synthesis of oxygen-free graphene for doped in titanium dioxide TiO₂. This work hypothesised the introduction of a new method for incorporating graphene nanoplatelets GNP in Anatase TiO₂ using adhesive nanocomposite material, which has been done to enhance the conductivity of the nanocomposite. This work also argues with lamina problems in Graphene oxide, which reduce electron mobility and cause the electron pathways to be rerouted. The characteristics of the nanocomposite measure the colour difference, the photocurrent-voltage measurement (I-V measurement), Raman Spectroscopy, and Energy Dispersive Spectroscopy EDS. Simple visual observation results for various thin films show a colour shade difference due to the better dispersion of the nanocomposites. The uniform colour change with different weight ratios can also show the distribution of graphene sheets. Similarly, similar ratios to photocurrent-voltage readings were obtained by the different nanocomposite weights in I-V measurement. The Raman spectroscopy also recognises the existence of well-composed 2D energy band GNP sheets cooperated inside the TiO₂. Finally, the work concludes with the reduction of the oxygen in weight ratios atomic, which lead to a better atomic level and the optimal weight ratio of GNP sheets to Titanium to increase the free mobility of electrons.

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1. INTRODUCTION

1.1. Background of the Study

In his 2003 speech at Rice University, Nobel prize winner Richard Smalley listed environment and energy as one of the ten issues facing humanity [1]. He stressed the high demand for sustainable energy sources. Today, the human population growth and the ongoing rapid increase in demand for energy have resulted in an environmental crisis because our energy demands are dependent on fossil fuels and generate greenhouse gas. Renewable sources such as biomass, hydropower, geothermal, tidal, ocean waves and wind energy have nevertheless received much attention. Furthermore, a significant focus was placed on solar energy due its abundance. However, the high cost of manufacturing and low market share for the 2nd generation silicon based solar cells has slowed the investment for such a formatable energy source, Furthermore, after O'Regan and Gratzel's pioneering work in 1991 [2] Who published the paper introducing low-cost, high-efficiency solar cell development (DSC's or DSSCs) devices I which started the 3rd generation solar cells, in comparison with 2nd photovoltaic silicon devices, these devices use low cost and possible non-environmentally hazardous materials and processes.

1.2. DSSCs

The DSSC is designed of three main parts (photoelectrode, electrolyte and counter electrode) and a sandwich of two conducting glass substrates. [3]. The use of semiconduction such as Tin oxide (SnO₂), titanium dioxide (TiO₂) or zinc oxide (ZnO), which acts as scaffolding material in dye molecules, also provides a transmission medium for electron charge when electrons reach the conduction band. [4]. However, due to its superior properties, such as low toxicity, high chemical stability and mesoporous microstructure, TiO₂ is considered the most useful and practical semiconductor. Moreover, anatase (tetragonal) is the most used from three primary crystalline forms of titanium because of its high electron mobility [5, 6]. However, TiO₂ only absorbs photons in the ultraviolet light region and has a high speed of recombination problem with electron-hole recombination. [7]. Illuminating light to the cell can stimulate the electrons in the dyes and promotes electrons from Highest Occupied Molecular Orbital (HOMO) to the Lowest Unoccupied Molecular Orbital (LUMO), which also improves the conduction band (CB). [4, 8].

A significant influence affecting device performance is a modification of the DSSCs photoanode by doping, morphology and by changing the thickness [5]. A variety of materials based on noble metal elements have been evaluated to enhance the performance of TiO₂. The superior candidates are Gold based (Au-NPs) [9], since gold acts as a high-efficiency dye sensitizer for typical plasma-induced enhancement of light absorption giving rise to a high photocurrent. A gold-nanostar morphology is employed (Au- nanostars) to enhance photovoltaic performance light absorption, [10], that improves performance in DSSCs due to its anisotropic properties. Another study realises Silver Nobel Metals (Ag-NPs) [11] formed as nano-composite photoanodes to enhance light absorption attributed to Surface Plasmon Resonance (SPR). Furthermore, that study of Ag-NPs deposited on titanium nanotubes TNT arrays demonstrates a higher conversion rate without Ag-NPs. However, most studies review rare earth element dopant materials, which are prohibitive for mass-production due to processing cost.

In 2004 two scientists Andre Geim and Konstantin Novoselov discovered Graphene [12]. A capable material that has exceptional superconducting characteristics in one atom. Those capabilities inspired many potential new ideas for research that focus on device efficiency. Partially because of the oxygen reaction medium, it is, of course, an oxidising agent, notably since the efficiency of Graphene in photocatalyst activity can be improved with Anatase rather than Rutile [13]. Moreover, comprehensive research has been explored as a photoanode enhancer concerning graphene, graphene oxide and many other types of carbon. Carbon as non-metallic anions was mostly incorporated into the DSSCs, the redshift of carbon-doped TiO₂ as inspected in the UV / Vis spectrum, and the XPS measurement indicates a reduction in the band gap resulting in higher conductivity, as can be observed on Jsc, and the intensity of electron transport as shown by improved lifespan [4]. Wang et al. performed a three-step technique of forming a TiO₂ porous photoelectrode model using graphene oxide. Due to the sheet formation of the graphene into the titanium framework, the TiO₂-GO has improved the conversion efficiency and formed a porous framework. [14], Modified hummer method has been implemented in another research by Wang and Cho [15], to decrease GO to rGO. The situation of the research is to design three layers of TiO₂-rGO film which include several proportions of the graphene doped in the TiO₂ for the DSSC photoelectrode as GR or rGO could be acquired by using an autoclave as a result of the decrease of GO. The UV-vis absorption spectrum results in an excessively elevated or low graphene content which was not desirable to adsorb dye. An adequate material of decreased graphene can make TiO₂ nanoparticles more evenly distributed. A Peiris et al [16]. stacked rGO-TiO₂ study initialized by the method of electrophoretic deposition (EPD) to position stacked film alternative rGO-TiO₂ photoanode in a set electrode layer thickness. Due to better current short circuit with the Graphene, which acts as a bridge to the FTO glass substratum, the overall interlayer incorporation TiO₂ has achieved 40% higher efficiency than the standard cell meeting the same conditions. In addition, a greater amount of stacks have resulted in a reduced present short circuit owing to serious interface changes that generate more routes to unwanted instructions. In a research on titanium (IV) isopropoxide (TTIP) with distinct wt percentage ratios of graphene oxide reduced(rGO), one step sol precipitation peptization method was implemented while using two kinds of dye, well-known N-719 (synthetic dye) and natural green chlorophyll (organic dye). The resulting effectiveness is 3.95 percent for the TrGO-2 0.03 g wt ratio and 0.67 percent for the natural green day [17].

Many studies on bandgap reduction by cooperating TiO₂ with graphene oxide GO or reduced graphene oxide rGO composite has shown that the bandgap of the non-metal oxide is reduced while the electron life spin improves. Oxidation of graphene, however, increases the distance between graphene sheets due to the trapping of oxygen. [18]. Moreover, the polarity of the graphene layers is improved, thus increasing water solubility. Moreover, researchers addressed this issue by applying an upgraded method to the conventional hummers' method to reduce the oxygen level, thus: reducing the insolation problem in the graphene oxide matrix.[15]. In GO, oxygen levels in graphene were respectively 31.31% to 68.69%. Moreover, even after 1440 minutes of sodium borohydride (NaBH₄) as a reducing agent, the decrease method

was not very significant, changing the percentage values for the oxygen in graphene oxide from 84.80 to 12.35. [19].

1.3. The Objective of this Study

This study propose a new method for decorating/ doping graphene nanoplatelets GNP in to pure anatase phase of titanium oxide TiO₂ to enhance the electron mobility in the conduction band TiO₂. The strategy of the method is to ensure the correct dispersal of metal oxides and the powder form of graphene, achieving an extremely reliable weight ratio and reducing time and afford of synthesising the nanocomposite using convnsional methods.

2. RESEARCH METHOD

2.1. Materials

Titanium (IV) Oxide, Anatase Nanopowder, <25 nm Particle Size, 99.7 Trace Metals Basis. Graphene Nanopowder: AO-3: 12nm Flakes-100g, Graphene Supermarket. Ethanol 95% Denatured, Hamburg C0312, 2-Propanol for Analysis Emsure, ACS, ISO. Acetone of Analysis Emsure, ACS, ISO. Acetic Acid, Alpha-Terpineol, Technical Grade, 90%.

2.2. Aim of Work

This proposed method is designed to measure the powder-like doping weight ratio between TiO₂ and GNP. This takes place with the help of the precious amount of adhesive materials, dilutions and synthesises the nanocomposite. A density equation is, however used for volume conversion and vice versa, as mentioned below:

$$P = m/v$$

The density (**P**) is the material's density. In addition, mass (**m**) is the material divided by gravitational acceleration (gram). And meanwhile, the volume (**v**) is the material space (milliliters). In contrast (Table 1), the ratio of materials used in this experiment method due to the weight of TiO₂ /GNP elements.

2.3. Synthesising TiO₂/GNP

As previously conducted in our work [20] detailed in **Error! Reference source not found.** First beaker, the binder is produced in a weight ratio (1:10) beaker by mixing ethyl cellulose into ethanol and then magnetically stirred for two hours. In the meantime, the volume of ethanol is reserved for use on a weight / Volume ratio of (17:1) of (TiO₂/GNP powder). Second beaker, is ultrasonic with the required total amount of graphene powder with the ratio (1:13) of the already reserved ethanol for (2 hours), ensuring graphene sheets are dispersed and separated. There is a sequence of steps in place to ensure that the graphene powder is synthesised with the semiconductor (TiO₂) for the preparation of the dilute. Starting with Acetic Acid at DI water and TiO₂ at Table 1 ratios then adding the remaining ethanol to the beaker gradually while stirring.

Next, the graphene powder is added to and stirred into the diluted TiO₂. In addition, a further 30 minutes of ultrasound is needed to ensure the dispersion of graphene into metal oxide before the addition of α-terpineol, which is then stirred for another 60 minutes. Finally, the ethylcellulose beaker is added to the TiO₂ /GNP nanocomposite and repeatedly stirred for an additional eight or ten hours. Finally, after overnight stirring, the evaporation process will commence with heat exposure of 80 to 110 °C depending on the concentration of graphene.

Table 1. The Calculation of TiO₂/GNP Nanocomposite

TiO ₂ /G (gram)	DI water (gram)	Acetic Acid (gram)	α-terpineol (gram)	Ethanol (gram)	Stirring Speed
1	0.8	0.16	3.3	17	500
2	1.6	0.3	7	34	500
3	2.5	0.5	10.7	51	1000
4	3.3	0.6	14.5	68	1000
5	4.1	0.8	17.7	85	1500
6	5	1	21	102	1500

- a. The weight ratio of the Ethanol does not include the volume of ethanol that is already used to make the Acetic Acid paste
- b. Most of the chemical materials calculated in (gram) to ensure presence measurement

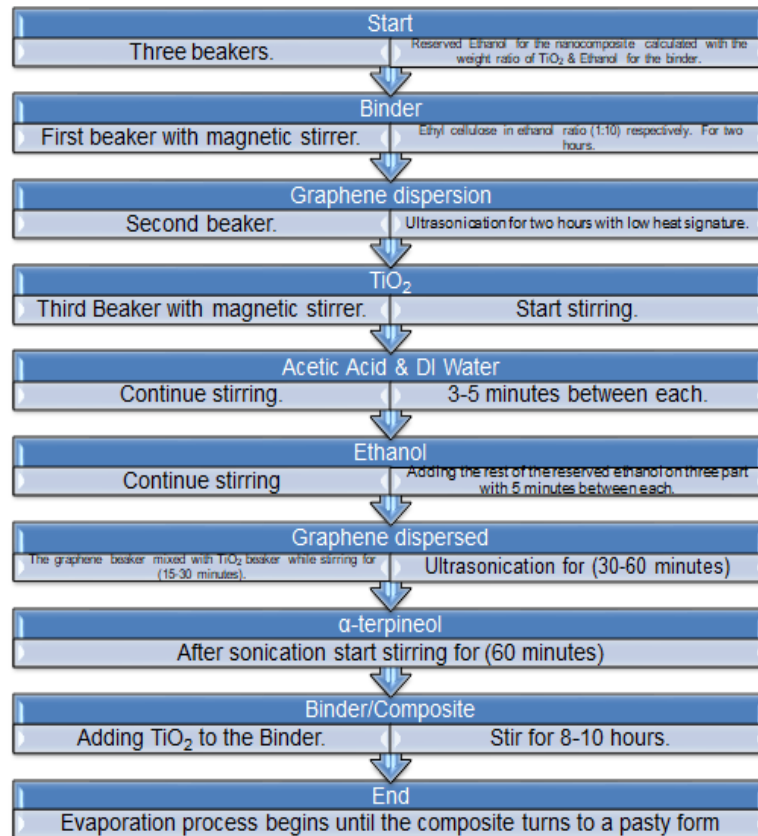


Figure 1. The proposed method of synthesising the nanocomposite of GNP and TiO₂

2.4. Thin Film

The 2.5 x 2 cm substrates are rinsed with acetone, isopropanol, and ethanol and sonicated for 15 minutes with each solvent, respectively. Then they are dried on a heat plate. A “Dr Blade” method is used to deposit the paste as a thin film on the substrates. These are baked in the furnace for 30 minutes at 450° C in the air to activate and crystallise the titanium.

2.5. Instruments

This analyses the TiO₂ / GNP doping and carbon and oxygen levels in the thin film, in order to determine increased mobility of electrons. Also, different weights of the graphene-titanium nanocomposites will be checked with the same percentage ratio. Raman spectroscopy is used to define the resulting thin film microstructure's structural fingerprint. The atomic structure of nanocomposite is determined by this method. The molecular weight of carbon and titanium concerning oxygen is also observed by Energy Dispersive Spectroscopy (EDS).

3. RESULTS AND ANALYSIS

3.1. Characterisation and Reliability Test

In Figure 2. Demonstrates the evident graphic doping efficacy of the TiO₂ film after rinsing synthesised composites of various levels. This begins at 0% graphene or pure TiO₂ then 0.2%, 0.5%, 1%, and 1.5% graphene from A to F, respectively. Each stage of the concentration shows a colour difference. The colour spectrum begins from bright white with a pure TiO₂ film as shown in the original film (A) and has finished in Gray-like as shown in (B, C, E, and F). The shift in colour is due to the uniform graphene concentration mixed in TiO₂. In comparison, Figure 3. Shows the photocurrent measurement (I-V measurement) performed as a credibility test as two composites with the same graphene doping percentage as in 1 g and 2 g of the nanocomposite, Those results indicate the same voltage-current density expressed by (V) and (mA), respectively.). Therefore, the results of the last test were highly considerable since they have similarity in Fill Factor, efficiency, and Voltage-Current as the other weight nanocomposite.

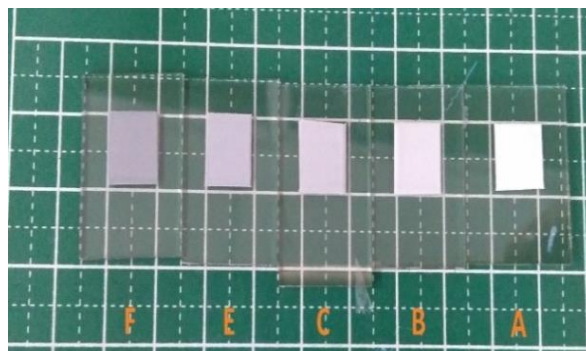


Figure 2. GNP doped into TiO₂ with different concentration starting from Pure TiO₂ (A) to higher concentration of graphene in (B, C, E, and F), respectively

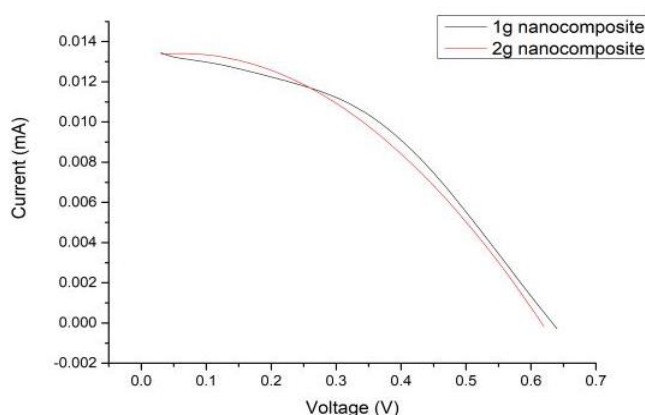


Figure 3. The measurement of two different composite with same Graphene ratio to confirm method credibility, The Blue and orange lines show nanocomposites with the same doping ratio of Graphene but differ in Titania & Graphene weight. They have almost the same Current Voltage Curve.

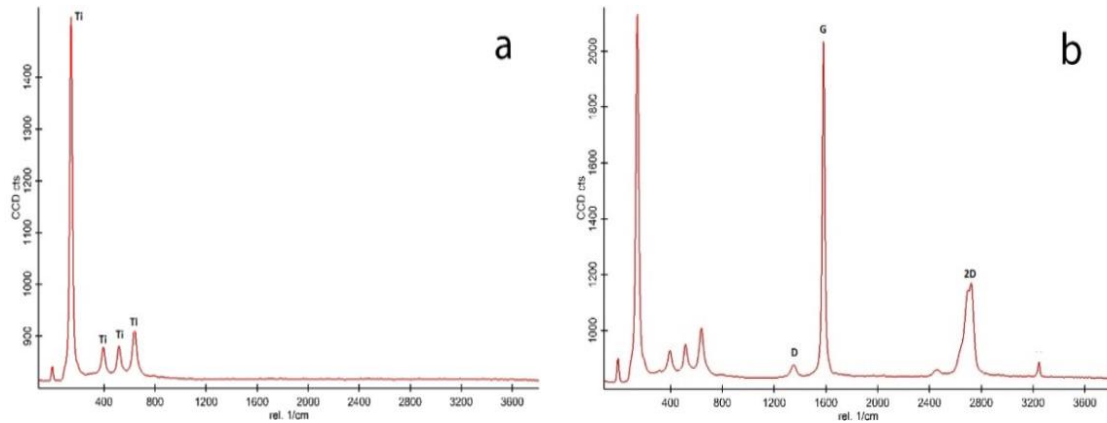
3.2. Raman Spectroscopy

The graphs presented in Figure 4 outlines the Raman spectroscopy results on two of the post-annealed TiO₂ synthesised paste substrates, which were both deposited using a Dr Blade Technique. Figure 4 (a.), outlines peaks in the test sample of bare TiO₂ around 150 to 500, which is an anatase fingerprint.

Furthermore, Figure 4 (b.) shows Raman spectra of the TiO₂/GNP doped solar cell. The sharp peaks reflect the film's crystallinity. The spectra show three sharp peaks at G, 2D, and D bands from 1300cm⁻¹ to 2750cm⁻¹. The D band indicates quasiparticles and collective exchange that can be represented by free particles like electrons and photons, which indicates high free electron mobility on the graphene side. The sp² phonon vibrations cause peaks in the broad G band. The 2D band represents the fingerprint for Graphene as a nanocomposite with the G band confirming a multi-layered graphene flex [21].

3.3. OriginPro

In Figure 4 (b). The 2D peaks, it demonstrates the formation of nearly a single-layer of graphene represented by 2678 cm⁻¹ and above 10 cm⁻¹ or multi-layered graphene represented by 2697 and above 20cm⁻¹ [22]. Finally, to determine the subtle presence of graphene, an analysing program called OriginPro is utilised, which integrates the area under the intensity peaks. By comparing the intensity ratio of 2D/G peak in Raman Spectra derives a (9.53 ratio) of single-layered graphene sheets. However, the broadening shape of the 2D band is evident because of the Bernal stacking of layers, which is an ABA stack as shown in Figure 5.



(a) Raman Spectroscopy for a bare TiO_2 thin film the peaks indicate anatase phase of crystallization

(b) Another thin film with a composite of TiO_2 /GNP indicated both elementsfingerprint with a good trace of Graphene in (2D) band

Figure 4. Raman spectroscopy in respect to bare titanium oxide thin film and titanium oxide/ graphene nanocomposite

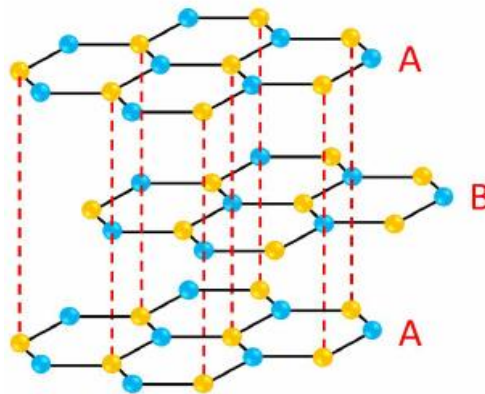


Figure 5. The ABA stacking of the graphitic layer in the thin film

3.4. Energy Dispersive Spectroscopy (EDS)

EDS measurements are performed on a $5 \mu\text{m}^2$ area, and the results are shown in **Error! Reference source not found.** A thin film of a nanocomposite of TiO_2 and Graphene sheets has been tested, several peaks associated with (Ti, C, O) are verified. Furthermore, in **Error! Reference source not found.** (a.), by the recognised peaks of current on the thin film, derives the weight ratio and the atomic level of the oxygen, which is lower than that of the study conducted that used Graphene oxide (GO) as a conductivity enhancer. As shown in **Error! Reference source not found.** (b.), this cell allows considerably more exciting free electrons to exit the thin film. [23]. Besides, as shown Table 2 gives a comparison of the results from this investigation related to other studies that use graphene oxide as a dopant. The unusual atomic level of oxygen shows that almost half of the entire circuit comprised of oxygen and confirmed a small level of carbon weight by ratio and at the atomic level. Another study involved in the table explains the results for TiO_2 /Graphene nanohybrids handled by nitrogen [24]. Although that study superseded in decreasing the Oxygen level, the Titanium level dramatically decreased to 7.40%. Plus, that high chemical method employed with higher firing temperatures ($540 \text{ }^\circ\text{C}$ - $700 \text{ }^\circ\text{C}$) Another a study of CQD [25, 26] which applied a TiO_2 photoanode to a DSSCs indicated a higher Carbon weight percentage, which is 2.94%, but still lower than this study, and the Oxygen level is within a comparable normal range of 36.26%, compared to 32.88% in this study.

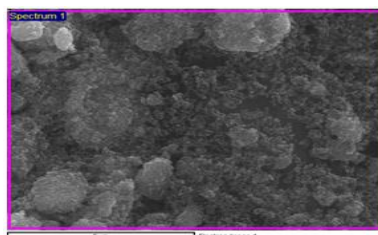
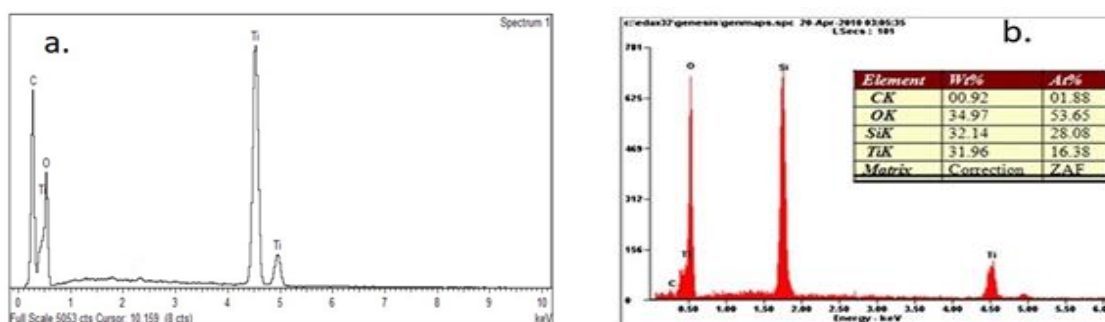


Figure 6. (5 μm²) as part of the sample of study that include (TiO₂/G) measured by EDS

Table 2. EDS Weight & Atomic Percentage Level

Element	Weight %	Atomic%	Weight% [20]	Atomic% [20]	Weight% [21]
OXYGEN	32.88*	40.95*	34.97	53.65	36.26
CARBON	25.00*	41.48*	00.92	01.88	2.94
TITANIA	41.86*	17.42*	31.96	16.38	60.80
SILICON	-NA	NA	32.14	28.08	NA

- a) The previous study of CQD did not provide the Atomic% of the EDS measurement.
- b) The previous study of Nitrogen-Doped TiO₂/Graphene Nanohybrids did not provide the weight percentage of the EDS measurement.



(a) measurement of our sample we can clearly see a higher peak for (Ti) and (C) compare to (O).

(b) a previous study for GO with lower carbon concentration compare to (O).

Figure 7. Energy Dispersive Spectroscopy (EDS) in respect to our nanocomposite structure and a previous study in that regard [20]

4. CONCLUSION

In summary, NanocompositeTiO₂/GNP efficiency in this research enhanced. The synthesis TiO₂ / GNP shows a significant shade of colour difference with different GNP ratios. In contrast, in colour difference with higher graphene concentrations, the various weight ratio between Titania and Graphene can be seen, indicating a consistent dispersion of the graphene particles. The I-V characterisation demonstrated credibility, as when the performance of multiple composites with the same graphene weight ratio is similar in photocurrent density characterisation. The results of Raman spectroscopy showed a 2D band for both single layer and multi-layer graphene to confirm the existence and performance of graphene within a photoanode. The spectroscopy also illustrates phonons in the electronic band structure that relate to high particles dispersal in the graphics layer.

Furthermore, The information assessment using the OriginPro software shows an expanding 2D band shape attributed to the graph layer stacks. The EDS (Energy Dispersive Spectroscopy) technique shows greater atomic mobility for both titanium and graphene, corresponding to a less oxygen-containing presence in a thin film. Since oxygen traps or redirects the electron, the mobility of the electron between the sheets is influenced by oxygen. The method used for producing devices also makes the creation of highly-functional composites time and effort cost-effective. The nanostructure also achieves an obvious decrease in oxygen, resulting in a better atomic level for graphene, which allows an improvement in photoanode layer electrons mobility.

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