

Study the Characteristics of the nanocomposite prepared by two steps laser ablation in liquids

* R. Sh. Alnayli

Zeena Hakim

University of AL-Qadisiyah, Department of physics, IRAQ .

* Corresponding Author E-mail: Raad.Anayli@qu.edu.iq

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ABSTRACT

In the present work, structural, optical, electrical and sensing properties have been studied for the (TiO₂/rGO) nanocomposite prepared by the pulse laser ablation method in the liquid, where the Nd-Yag laser was used, and the two wave pulses (1064-532) and 300mJ, which were deposited on glass bases , The structural properties have been investigated by X-Ray diffraction technique analysis and morphological by atomic force microscope (AFM), The particle size of the membranes is less in the films prepared using wavelength (1064) than in the prepared films using wavelength (532). The optical properties of the nanocomposites have been determined by using the optical transmittance measurements in the spectral region from (300-1100) nm. Electrical properties such as I-V properties was also studied. sensing properties measurements showed good Humidity sensitivity within the range (20-80) % RH.

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دراسة خصائص المترابك النانوي المحضر من خطوتين الاجتثاث بالليزر في السوائل

زينه حاكم

رعد شاكر النايلى

جامعة القادسية / قسم الفيزياء

الكلمات المفتاحية:

المترابكات النانوية
التشطية بالليزر في السوائل
TiO₂/rGO
الخصائص التحسسية

الخلاصة

في العمل الحالي تمت دراسة الخصائص التركيبية والبصرية والكهربائية وتحسسية للمركب النانوي (TiO₂ / rGO) المحضر بطريقة الاستئصال بالليزر النبضي في السائل ، حيث تم استخدام ليزر Nd-Yag ، والنبضات الموجية (١٠٦٤-٥٣٢) و ٣٠٠ مللي جول ، والتي تم ترسيبها على قواعد زجاجية ، تم دراسة الخصائص التركيبية بتحليل تقنية حيود الأشعة السينية والمورفولوجية بواسطة مجهر القوة الذرية (AFM) ، وحجم جسيمات الأغشية أقل في الأغشية المحضرة باستخدام الطول الموجي (١٠٦٤) مقارنة بالأغشية المحضرة باستخدام الطول الموجي (٥٣٢). تم تحديد الخصائص البصرية للمركبات النانوية باستخدام قياسات النفاذية الضوئية في المنطقة الطيفية من (٣٠٠-١١٠٠) نانومتر. كما تمت دراسة الخصائص الكهربائية مثل خصائص I-V. أظهرت قياسات خواص الاستشعار حساسية جيدة للرطوبة في حدود (٢٠-٨٠) % رطوبة نسبية.

1. INTRODUCTION

In recent decades, TiO_2 -based nanohybrids have conventional increasing interests from researchers for their great potential in the many industrial applications, such as solar cells, photocatalysts, and sensors [1,2]. Under the concern to alleviate the decline of the environment impurity and energy crisis and to protect sustainable global growth, It is an imperative n-type semiconductor with has band gap of 3.2 eV, there are two types of crystal structures which consist of anatase and rutile phase [3]. Among dissimilar materials that can be certain to make composites with TiO_2 , carbon materials deal unique benefits, such as constancy and chemical inertness, and tunable textural, and chemical assets [4,5]. Graphene is one atomic layer thick 2D material. It has unique features such as great essential carrier mobility, High theoretical surface area, electrical conductivity and high thermal [6]. Nanocomposite materials having TiO_2 are more striking than bare TiO_2 [7]. Henceforth, modification TiO_2 with graphene might surpass the limitations of both materials with their synergistic effects and show the well sensing properties.

TiO_2/rGO was synthesis for the first time by Williams *et al.* [8] through UV-assisted photocatalytic reduction method. Since then, several retreats of methods are being followed to synthesize such material of interest in various applications photocatalysis [9], hydrogen generation [10], gas sensing [6]. Thus, graphene oxide has to be reduced to return the exclusive properties found in the pristine graphene. the production from the reduction process could be added modified and used for a wide series of applications [11]. (GO) has appeared as a forerunner for bulk production of graphene-based materials, as it can be produced from cheap graphite powders [12] and usually covers groups "hydroxyl and epoxide" on their basal

plane, "carbonyl and carboxyl" groups on their edges [13]. A simple technique is described for reduction of GO solution by pulsed laser ablation which majority of oxygen-containing functional groups of GO are removed. The reduction time was carried out fast at room temperature. This method offers an active way to production r-GO in short time and care for environment [14].

2- Experimental Work

Titanium Dioxide TiO_2 NPs were synthesized by laser ablation of in height purity Ti target placed in double distilled water (DDW) at room, temperature. Ti target is located in the bottom most of quartz vessel complete with 4mL of liquid and exposed with Q-switched, (Nd:YAG) laser worked at wavelength of (532,1064)nm, duration of pulse 7ns, and repetition frequency of 6Hz. The energy of laser was used to ablation Titanium target was 300mJ/pulse, the time of ablation was: 6min. The beam of laser was fixated on (Ti) target using focusing lens of 100mm focal length. TiO_2 nanoparticles produced by the above step (by PLAL), were mixed with (1mL) of GO (<450 nm, Cheap Tubes) (0,00625g each 1mL of deionized water), we denoted (TiO_2/rGO ,532) for the sample prepared with a wavelength of 532 and (TiO_2/rGO ,1064) for the for the sample prepared with a wavelength of 1064, This suspension was irradiated for (10 minutes) with the similar the parameters of laser, Through this method, GO becomes reduced rGO and concurrently, the nanoparticles TiO_2 anchor on the rGO sheets to formula TiO_2/rGO nanocomposite.

3- Resulting and discussing

3-1 X-ray diffract (XRD)

Figures (1),(2) describe films diffraction Graphene Oxide, and Dioxide Titanium-reduced graphene oxide (TiO_2/rGO) that prepared by pulsed laser ablation in Double distilled and

deionized water(DDDW) on cover glass substrates. X-ray diffraction test exhibited a high-intensity high peak for the Graphene oxide at the surface (001) and angle ($2\theta=11.85^\circ$) this agree with card (JCPDS Card NO.75-1621) this agree with researcher[7]. The results of X-ray for TiO_2/rGO are shown in Figure (2).

It had two peaks (002)(101) corresponding to degrees (10.80)(25.28) respectively ,graphen oxide in in (TiO_2/rGO) had polycrystalline structure with a hexagonal type crystal structure.

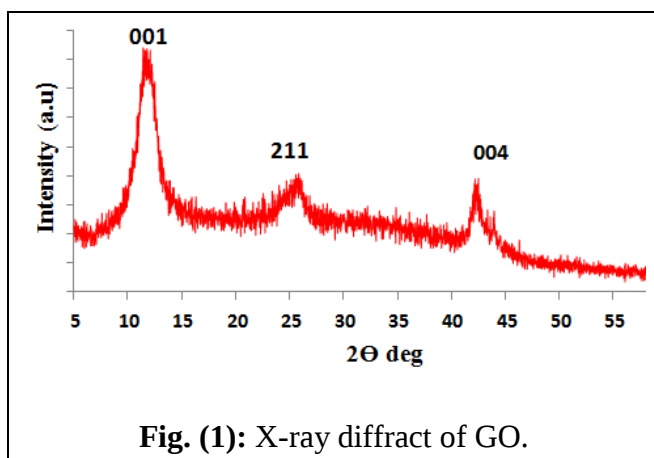


Fig. (1): X-ray diffract of GO.

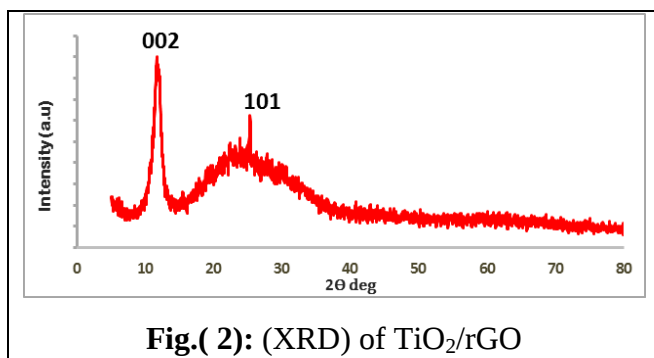


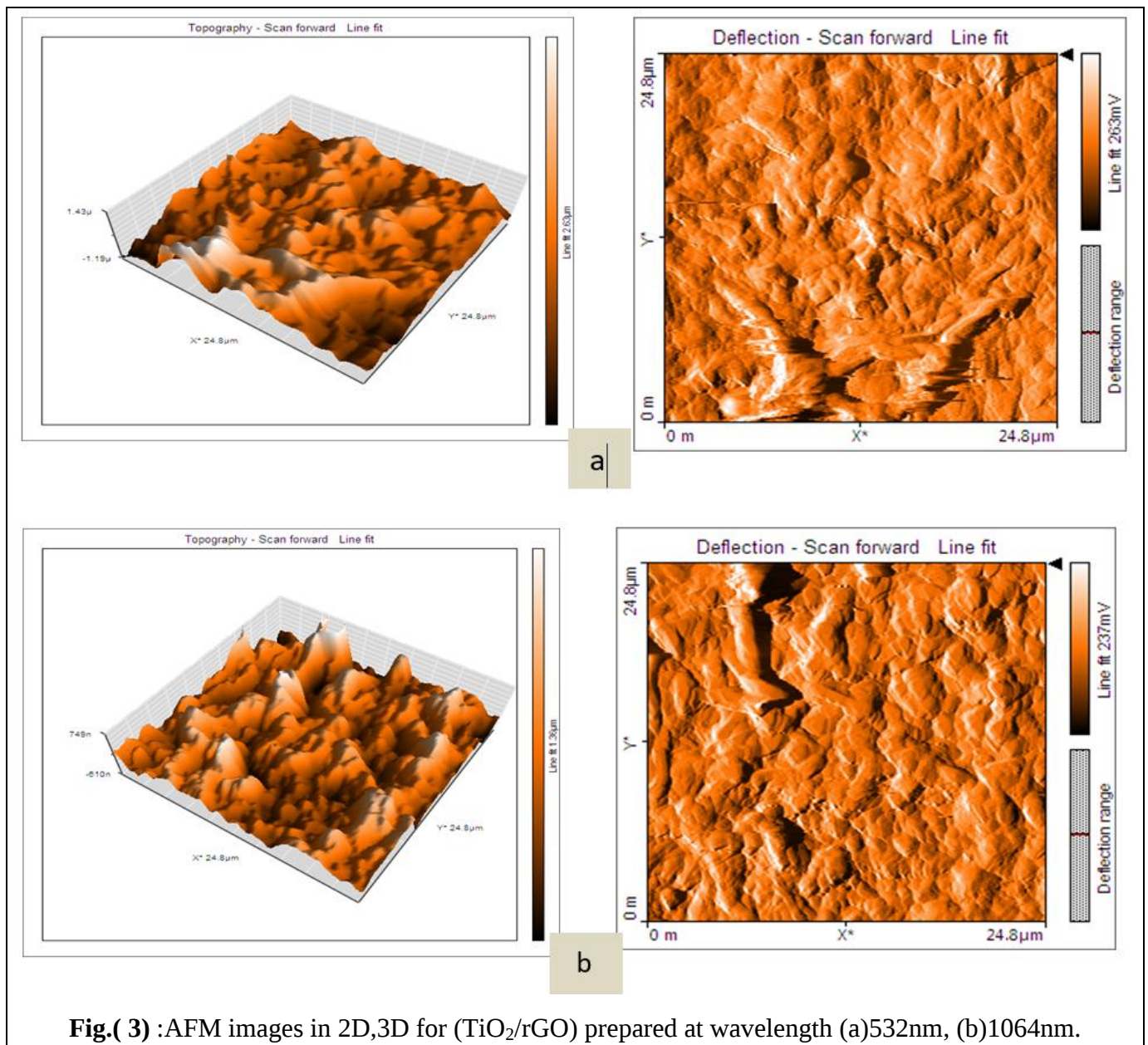
Fig.(2): (XRD) of TiO_2/rGO

3-2 Surface Morphology

Figures (3), (4) show structures of samples in two and three dimension for the ($\text{TiO}_2/\text{rGO},532$)($\text{TiO}_2/\text{rGO},1064$)nanocomposites produced using PLAL method at fixed energy 300mJ , wavelength (1064&532)nm. The results showed that the film materials and through the two-dimensional and three-dimensional diagnosis are homogeneous to a certain extent and homogeneous vertical heights, noting that there are no spaces in the surface of the material. . All the images show homogeneous distribution with columnar structure. The AFM analyze showed that average grain size of particles decreases when laser beam wavelength used in preparation increases , The root mean square (RMS) ,grain size and average roughness of the films are listed in below table.

Table (1) show Morphological characteristics of (TiO_2/rGO) prepared at wavelength 532nm and 1064nm.

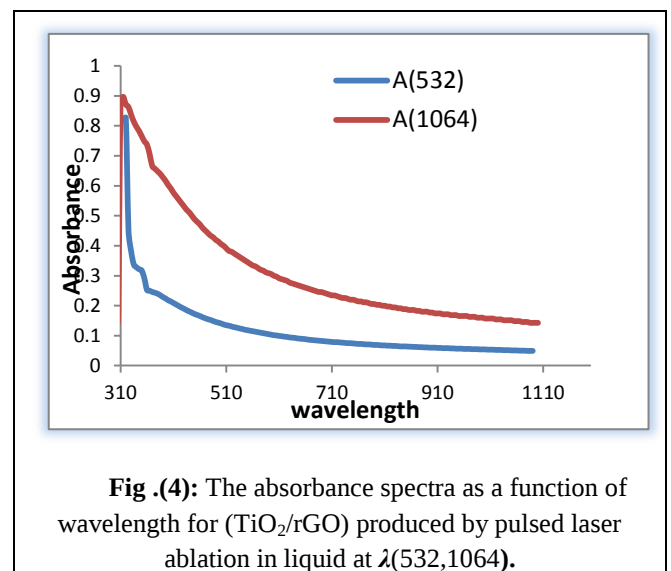
Sample	Root mean square Sq (nm)	Roughness average Sa (nm)	Mean grain size (nm)
($\text{TiO}_2/\text{rGO},532$)	375	284	68
($\text{TiO}_2/\text{rGO},1064$)	248	190	61



3.3 Optical properties

from the figure(4) It can be noticed that the absorbance for nanocomposites have a high values at wavelength in the neighborhood of the fundamental absorption edge (300nm) , then the absorbance decreases with the increasing of wavelength. In general, the absorbance of nanocomposites has low values in the visible and near infrared region this agree with[7] . The sample prepared at 1064 wavelength has the highest absorption of the sample prepared with wavelength 532. The value of the optical band gap that obtain off the linearly parts of the $(\alpha h\nu)^2$ as a functions of the photon energy $h\nu$

is (3.1) for TiO₂/rGO,532 and (3) for TiO₂/rGO,1064 .



3-4 Current – Voltage (I – V) characteristics

Figure (6) show the relationship between the current passing through (TiO₂/rGO,532) (TiO₂/rGO,1064) nanocomposites prepared by using wavelength(1064&532)nm and the voltage applied , , the relationship between current and voltages for (TiO₂/rGO,532) is linear and indicating the behavior of ohmic material and the Resistance can be calculated by Ohm's law where it's almost constant . while (TiO₂/rGO,1064) nanocomposite exhibit the behavior of diode material. the current flowed in the sample prepared by wavelength 532 wavelength shall be less than the current flowed in the sample prepared by the wavelength 1064.

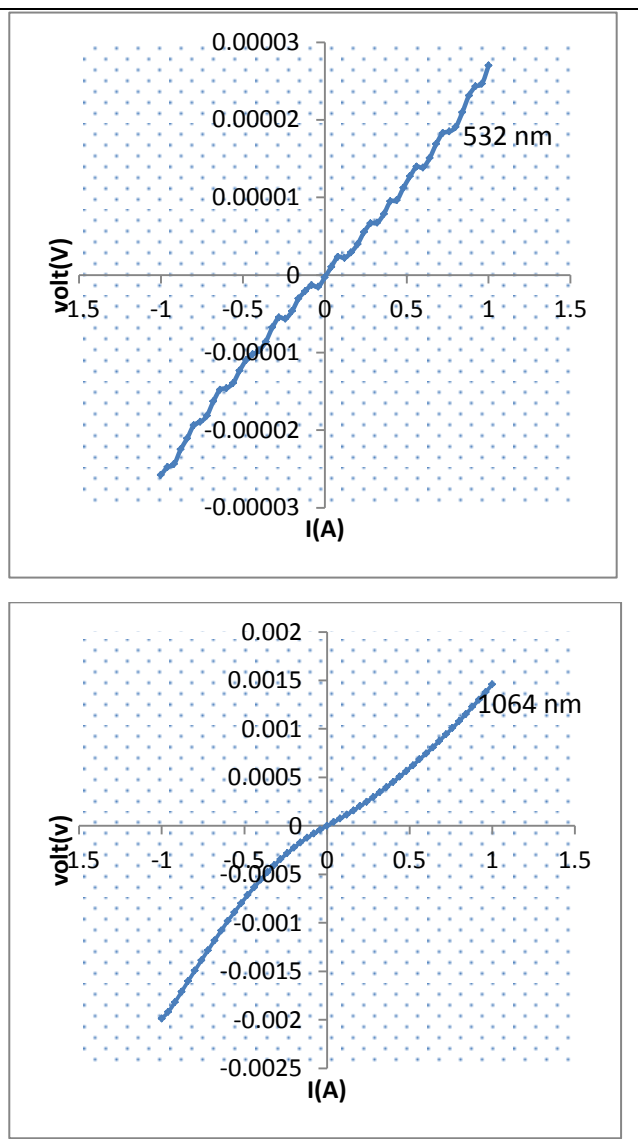


Fig.(5):): I-V characteristic of (TiO₂/rGO) nanocomposites

3-5 Application of (TiO₂/rGO) nanocomposite as sensor Humidity

Air in the atmosphere containing water vapor is named as atmospheric air. It is expedient to treat air as a mixture of dry air with water vapor since the structure of dry air remain relatively constant[15]. Calculating and monitoring humidity is economically essential for maintaining equipment and machinery used in medicinal, agricultural, engineering, and food stowing sectors; also, it is central for maintaining well and comfy living environments[16]. The greatest usually used terms are Relative Humidity, (RH) that definite as:" ratio of the amounts of wetness content of air to the full (waterlogged) vapor level that the air can grasp at a same known temperature ,pressure of the gases"[17].

The sensitivity of the sensor is definite as" the variation in forward current at a sure voltage of the sample in definite humidity with respect to its current at the similar voltages in dry conditions". Thus, the sensitivity is designed from the following equation [18].

$$S(\%) = \frac{I_H - I_{air}}{I_{air}} \times 100\% \quad \dots \dots \dots (2-27)$$

Figure (7) shows the response and recovery characteristics of the fabricated humidity sensors for (TiO₂ / rGO,532) (TiO₂ / rGO,1064) nanocomposites prepared by using pulsed laser ablation in DDDW at wavelength 1064&532 when they are exposed to 20% RH and 80% RH, respectively. the resistance decreases for all samples of nanocomposites with increasing the humidity and the nanocomposite have high sensitivity at high humidity, this behavior can be explained as follows: titanium dioxid reduced graphene oxide mixed with water show higher sensitive humidity sensing at room temperature, In the presence of humidity, it attracts – OH group hence the resistance

decreases. (TiO₂/rGO,532) and (TiO₂/rGO,1064) nano composites are porous in nature and has surface oxygen atoms which essentially arise attributed to the samples preparation technique. the nanocomposites adsorb the humidity, its resistance decreases due the increase of number charges of carries, the adsorption of water on the surface of the nanocomposites leads to the dissociation of hydrogen ions. These hydrogen ions are bonded to the surface lattice oxygen atom and forms the hydroxyl groups, this result agree with [16].

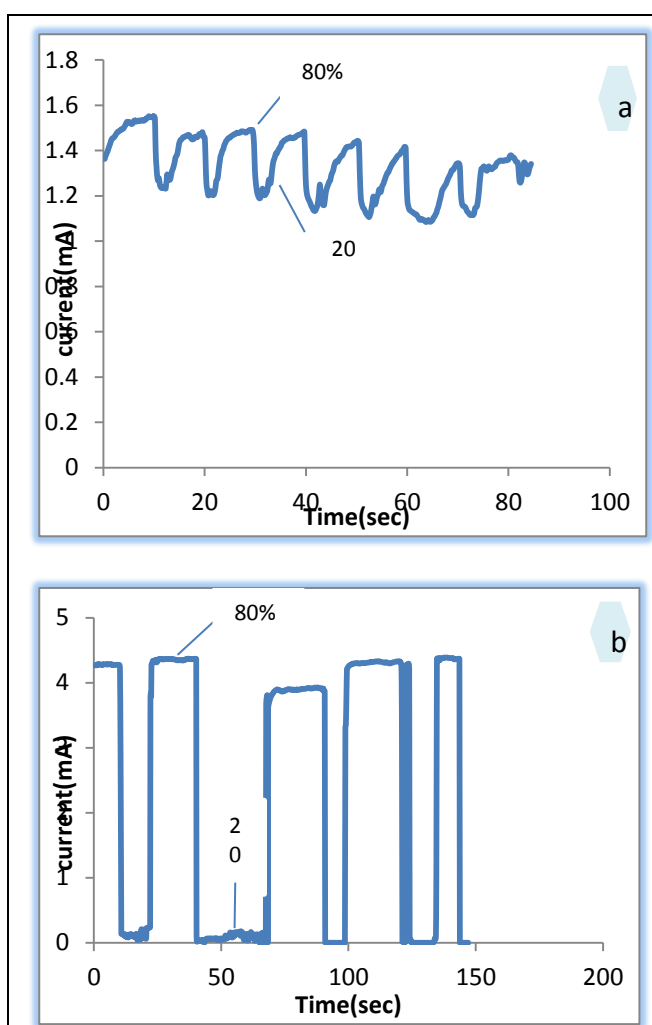


Fig.(6): Response and recovery characteristics of (TiO₂/rGO) prepared by using pulsed laser ablation in DDDw at a) 1064 nm b) 532 nm based sensor measured for 20 and 80 RH at 1V.

Table (2) shows the response time, recovery time and sensitivity of (TiO₂/rGO,1) (TiO₂/rGO,2) to 20 and 80 RH.

Sample	Response Time	Recovery Time	Sensitivity (%)
TiO ₂ /rGO,1064	2.72	3.12	16
TiO ₂ /rGO,532	0.39	2	98

We note from the figures that (TiO₂/rGO,532) nanocomposite had the highest sensitivity and the least response time and recovery time when they are exposed to low humidity 20 and high humidity 80% RH.

Conclusions

TiO₂/rGO nanocomposite was prepared by laser ablation method at different wavelength (532nm, 1064nm), and 300mj. The structure of TiO₂ in XRD investigation is anatase titanium Dioxide. The results of X-ray for (TiO₂/rGO) show that it had two peaks (002)(101) corresponding to degrees (10.80)(25.28) and graphene oxide in (TiO₂/rGO) had polycrystalline structure with a hexagonal type crystal structure.

AFM analysis of (TiO₂/rGO) showed that average grain size of particles decreases when laser beam wavelength used in preparation increases. UV-Vis spectrophotometer measurements The sample prepared at 1064 wavelength has the highest absorption of the sample prepared with wavelength 532. The I-V curve characteristic of TiO₂/rGO nanocomposites demonstrated behavior of ohmic and diode materials. And the current is increasing at high relative humidity for (TiO₂/rGO) nanocomposites. The best result recorded for sensitivity at 98.

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