SYNTHESIS, STRUCTURAL AND OPTICAL PROPERTIES OF ZnO-TiO_2-GO NANOCOMPOSITE

Su Su Lwin[1], May Hnin Thant[2], Than Than Win[3] and Yin Maung Maung[4]

Abstract

Graphene oxide (GO) nanoparticles were synthesized by Hmmmer's method and the synthesized graphene oxide was analyzed by X-ray diffraction (XRD), energy dispersive X-ray (EDX), fourier transform infrared spectroscopy (FTIR), UV-Vis spectroscopy and field emission scanning electron microscopy (FESEM). The XRD pattern reveled a (001) diffraction peak, signifying the successful synthesis of GO. The elemental composition of GO was characterized by EDX analysis. The mass ratio of C/O is 2.26. The results of FT-IR showed that C-O bond, O-H group with water molecules and C=C bond with graphene oxide. UV-Vis spectra of GO exhibited maximum absorption peak at 290 nm. And then, ZnO-TiO_2nanocomposite was prepared by simple mechanochemical activation method and the ZnO-TiO_2-GOnanocomposite was prepared by simple mechanical stirring followed by ultra-sonication. X-ray diffraction (XRD) technique was employed to examine the crystal structure and phase analysis of ZnO-TiO_2 and ZnO-TiO_2-GO nanocomposite. The elemental composition of ZnO-TiO_2 and ZnO-TiO_2-GOnanocomposite were characterized by EDX analysis. The optical properties of ZnO-TiO_2 and ZnO-TiO_2-GO nanocomposite were identified by ultraviolet-visible spectroscopy. UV-Vis spectra of ZnO-TiO_2 nanocomposite exhibited maximum absorption peak at 289 nm and 372 nm. For ZnO-TiO_2-GO nanocomposite, the maximum absorption peak are 543 nm and 645 nm. The particles sizes and the surface structure were examined by atomic force microscope (AFM) analysis and field emission scanning electron microscope (FESEM) analysis.

Keywords: Graphene oxide, Nanocomposite, Atomic force microscope, Hummer's method

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Introduction

Nanomaterial have found widespread applications in many devices including photo detectors, surface acoustic, wave devices, UV nanolaser, varistors, solar cells, gas sensors, biosensor, ceramics, nanogenerators, and field emission devices. However, the characteristics, especially the band gap of semiconductors are influenced mostly by the transition of particle size from bulk into the nanometer lever scale. Among the semiconductors, the conducting oxides (COs) (e.g. ZnO, SnO_2, TiO_2 etc.) have been researched and investigated the most for their possible use as photocatalysts. The organic dyes are notorious environmental pollutants which could be degraded under sunlight using the COs as photocatalysts. Among these different COs photocatalysts, TiO_2 and ZnO are important nanomaterials owning fascinating physico-chemical characteristics [Hameed Ullah et al (2014)]. Two metal oxides ZnO and TiO_2 are an important in terms of materials technology and have various applications in different industries [Majid jafan et al (2014)]. Zinc oxides of particle size in nanometer range have been paid more attention for their unique properties. They are widely used for solar energy conversion, non-linear optics, catalysis, varistors, pigments, gas sensors, cosmetics. Zinc oxide (ZnO) is an unexpansive, n-type semiconductor with a wide band gap having optical transparency in the visible range [S. Jurablu et al (2015) and P. M. Aneesh (2007)]. Nano titanium dioxide powder has many good functions and features, such as stable properties, non-toxic, high activity of photo catalysis, low cost and good at resisting chemical attack. It is also a nice photo catalyst, disinfector and antiseptic [Ziquanliu et al (2013)]. ZnO is a direct band gap semiconductor and TiO_2 is an indirect band gap with an energy band gap of 3.0 eV (anatase) or 3.2 eV (rutile) [Yuan Zhi-Hao et al (2001)]. To prevent the reduction of the zinc oxide, zinc oxide is combined with other metal oxides such as: TiO_2, Fe_2O_3, Al_2O_3, etc. In order to improve photocatalytic activity of TiO_2 and ZnO, a popular route is to form composite. ZnO–TiO_2 based sorbents appear to have the fewest technical problems. TiO_2 reacts with ZnO at high temperatures forming zinc titanates and form Zn_2TiO_4, $ZnTiO_3$ or $Zn_2Ti_3O_8$, depending on ZnO/TiO_2 molar ratio and on the preparation and calcination conditions.[Majid Jafan (2014)]. Graphene oxide is a wonder material, also offers new avenues in nano composites when combined with these wide band gap semiconductors. Excellent optical

transparency, mechanical strength and electrical conductivity have led to its use in numerous applications. Its chemical stability renders its use as nanocomposite with other materials. With its high optical transparency and specific surface area, it has been investigated with the aim of replacing indium based transparent conducting oxide, potentially opening way for flexible substrates [Muhammad Imran Ahmed et al and C. Pragathiswaran et al]. TiO_2, Fe_3O_4 and ZnO have been investigated as nanocomposite with GO, offering properties superior to individual materials [Wan-Kuen et al] ZnO-GO composites have been reported for applications like corrosion protection, photocatalysis, batteries, field emission prosperities and for incorporation in polymer solar cells [Muhammad Ali Johar et al**]**. In this study, ZnO-TiO_2nanocomposite was synthesized by ball milling at room temperature. A stoichiometric mixture of nano ZnO and TiO_2 powders in 1:1 molar ratio. The graphene oxide (GO) was added to the ZnO-TiO_2nanocomposite by simple mechanical stirring followed by ultra-sonication. The resulting powders were analyzed by XRD, EDX, FTIR, UV-Vis, FESEM and AFM.

Experimental Procedure

Preparation of Graphene Oxide (GO)

In this research, Graphite, Sulphuric acid (H_2SO_4), Sodium nitrate ($NaNO_3$), Phosphoric acid (H_3PO_4), Potassium permanganate ($KMnO_4$) and Hydrogen Peroxide (H_2O_2) were used as starting materials. The block diagram of sample preparation was described in figure 1. All chemicals were analytically pure and directly used as received without further purification. Graphene oxide (GO) was prepared according to the Hummer method. In detail, 500 ml beaker was filled with 108 ml of H_2SO_4, 5 g of graphite, 2.5 g of $NaNO_3$ and 12 ml of H_3PO_4 were added into the beaker. The suspension was stirred in an ice bath for 10 min (fig 2). Next, 15 g of $KMnO_4$ was added to the suspension. The rate of addition was carefully controlled to keep the reaction temperature below 5 ˚C and stirred in ice bath for 3 h (fig 3). Then the mixture was put in a 40 ˚C water bath for 60 min. The temperature of the mixture was adjusted to a constant 98 ˚C for 60 min while water was added continuously. The color of the mixture was changed into yellow. Deionized water was further added so that the volume of the suspension was 400 ml.

15 ml of H_2O_2 was added after 5 min. The reaction product was washed by rinsing and centrifugation with 5 % HCl (fig 4 & 5) then deionized (DI) water for several times to reach pH 5-7. Finally, the product was dried at 60 ˚C for 24 h in a vacuum oven. The obtained samples were characterized by X-ray diffraction (XRD) using (Rigaku RINT 2000) and energy dispersive X-ray (EDX). Fourier transform infrared spectroscopy (FTIR). UV-Vis spectrometer (UV-Vis; UV-1800) was used to measure the optical absorption properties of GO. The nano structure was confirmed by field emission scanning electron microscope (FESEM).

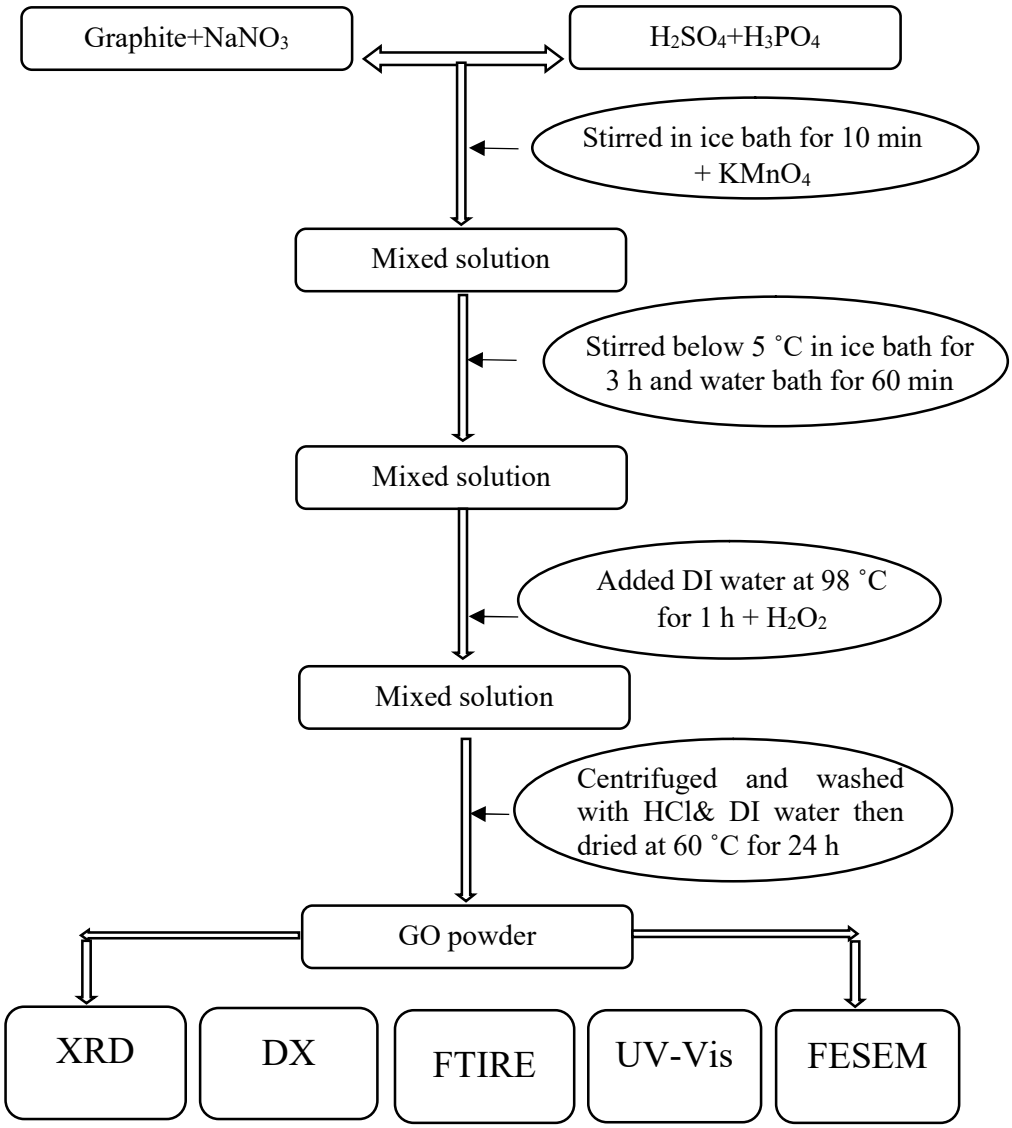


Figure 1. Block diagram of preparation of graphene oxide powder

Figure 2. Graphite, $NaNO_3$ and H_3PO_4 mixture was stirred in ice bath

Figure 3. Water bath

Figure 4. Centrifuge

Figure 5 . After centrifuging

Prepatration of ZnO-TiO_2nanocomposite

In this case, ZnO and TiO_2 were used as the starting materials. Firstly, ZnO and TiO_2 powder with stoichiometry ZnO: TiO2=1:1 molar ratio were mixed. Then the mixture was milled with the ball milling machine for 10 h to reduce the particle size. Three stage mesh-sieving was employed to get the uniform particle size. After the sample had calcined at 400˚C for 1 h. The block diagram of sample investigation was described in figure 6.The obtained sample was characterized by the X-ray diffraction (XRD) using (Rigaku RINT 2000) and wavelength dispersive X-ray spectroscopy EDX (Bruker Tiger S). Optical absorption spectra of the sample were taken with UV-1800 UV-Vis Spectrometer. The microstructure of the samples were examined by atomic force microscope AFM (Bruker N8 Rados) and field emission scanning electron microscope (FESEM).

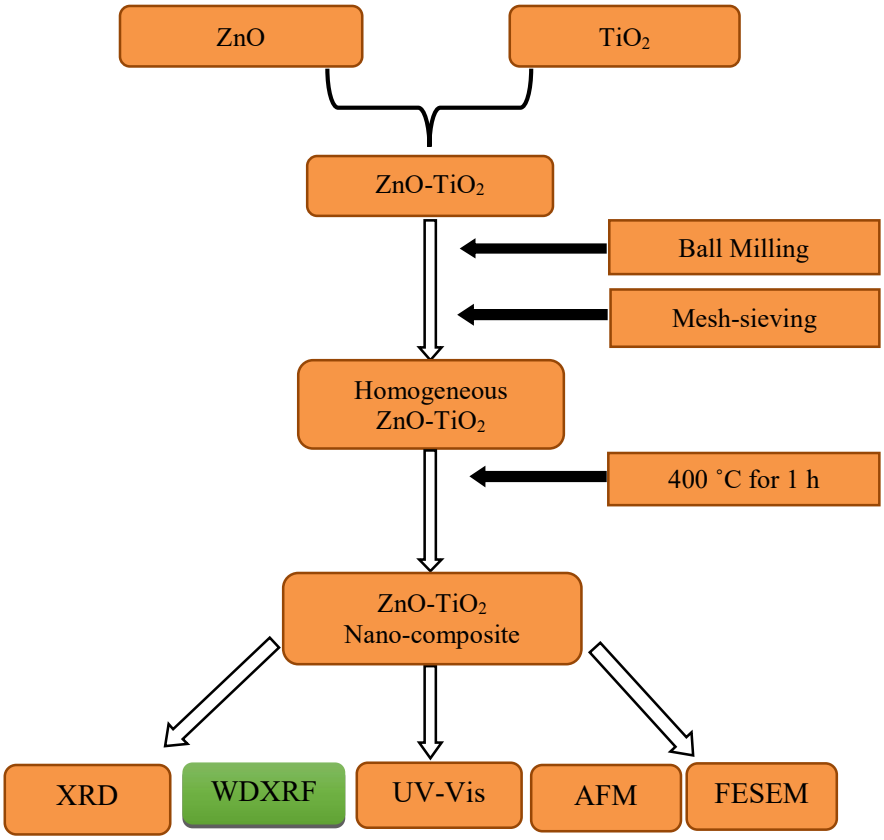


Figure 6 The block diagram of ZnO-TiO_2 nanocomposite

Preparation of ZnO-TiO_2-GO nanocomposite

ZnO-TiO_2-GO nanocomposite was prepared by added to the ZnO-TiO_2 nanocomposite by simple mechanical stirring followed by ultra-sonication. Firstly, 1 g of graphene oxide was added in 100 ml of distilled water and stirred by magnetic stirrer for 1 h. After stirring, the GO solution was ultrasonicated for 1 h. Then 2 g of ZnO-TiO_2nanocomposite was added into the solution and the mixture was ultra sonicated for 1 h again (figure7). The mixture was stirred by magnetic stirrer at 80 °C for 6 h. The mixture was then centrifuged and washed with ethanol for several times to remove the undecorated particle and unreacted chemicals (figure 8). Finally, the product was dried in an air oven at 80 °C for 6 h before characterization. Block diagram for the preparation of ZnO-TiO_2-GO nanocomposite are shown in figure 9.

Figure 7 Ultra-sonication

Figure 8. After centrifuging

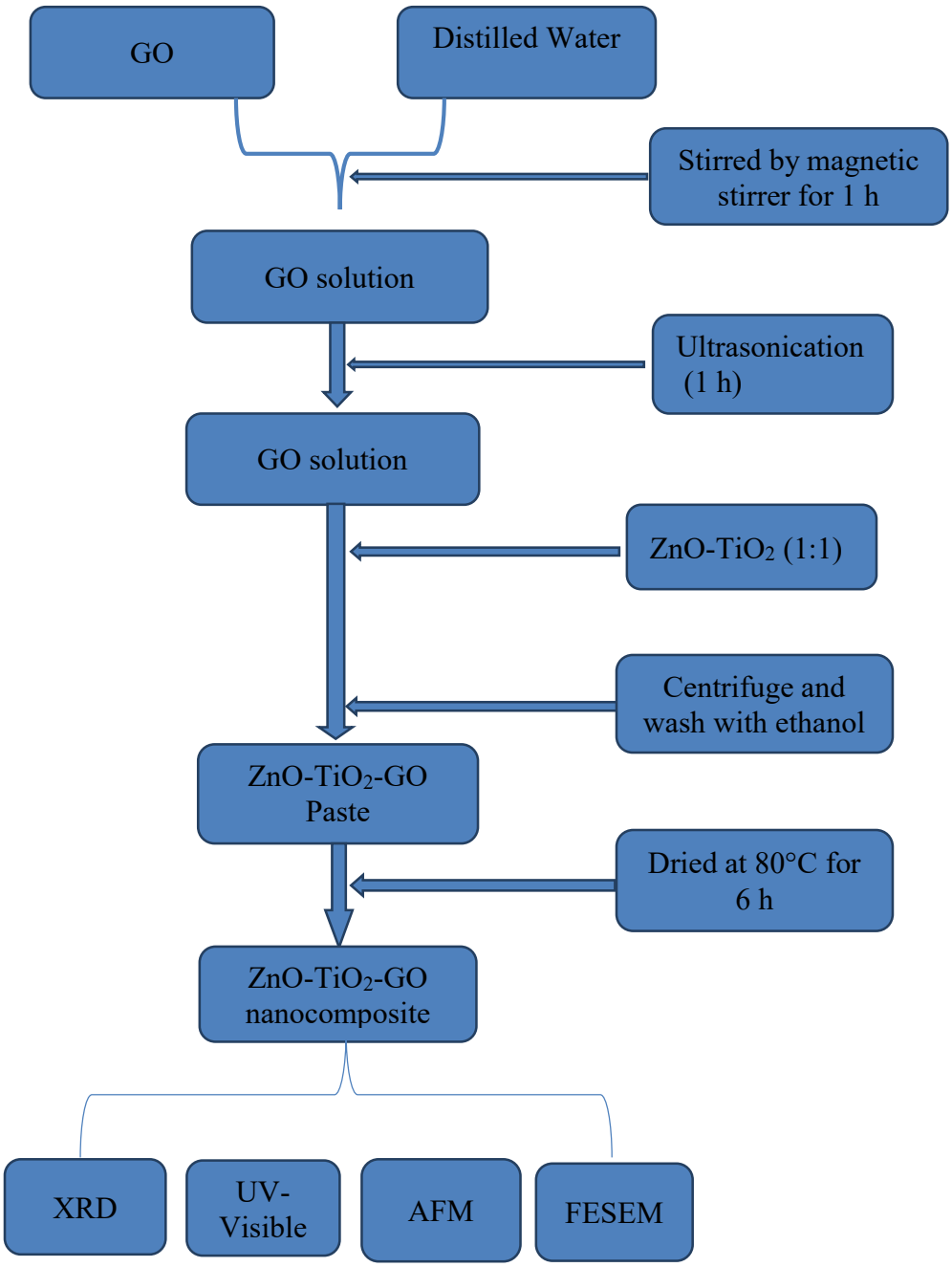


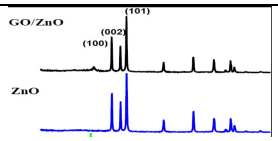
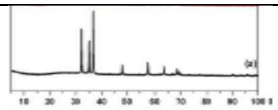
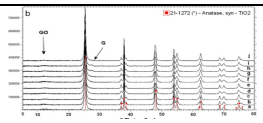
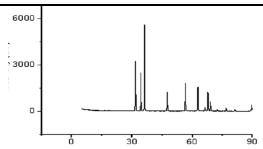
Figure 9. Block diagram for the preparation of ZnO-TiO_2-GO nanocomposite

Results and Discussion

XRD analysis

X-ray diffraction is used to determine crystallinity of polymeric materials. XRD uses the total X-ray scattering both the crystalline and amorphous phases to determine the crystallinity. The phase analysis of GO, ZnO-$TiO_2$and ZnO-TiO_2-GO powders were determined by using a X-ray Diffractometer (Rigaku RINT 2000). XRD was performed using monochromatic CuKα radiation (λ= 1.54056 Å) operated at 40 kV (tube voltage) and 40 mA (tube current). Sample was scanned from 5° to 70° in diffraction angle 2θ with a step-size of 0.02°. The XRD pattern of graphite, graphene oxide, ZnO-TiO_2 and ZnO-TiO_2-GO nanocomposite were shown in figures 10, 11, 12 and 13. According to the XRD pattern of graphite, which showed a strong and sharp diffraction peak at 2θ=26.61° has the interplanar distance of 0.334 nm. The graphene oxide were matched with standard library file 03-065-1528. GO shows an intense and sharp peak at 2θ=11.07° has the interplanar distance of 0.799 nm. The increase in interplanar distance of GO is due to the existence of oxygen functional groups. In figure 12, The TiO_2anatase (PDF-89-4921) and ZnO (PDF-89-0511) phased are observed in the X-ray spectra of mechaochemically activated ZnO-TiO_2nanocomposite samples with molar ratio of ZnO and TiO_2 as 1:1. ZnO-TiO_2nanocomposite X-ray patterns is present the higher crystallinity degree of investigated materials. In figure 13 the Graphene Oxide (GO) shows the diffraction peak at 2θ value of 11.07°. In composite the TiO_2 anatase and ZnO phased are clearly observed but the main diffraction peak of GO is absent and it probably lead to partial reduction of GO to graphene and a weak peak at 2θ = 26.50° appears (see in figure 13). It may be due to the low amount and relatively low diffraction intensity of GO in comparison with the diffraction intensity of ZnO-TiO_2 nanocomposite [C.Pragathiswaran et al]. Moreover the other possibility is due to the intercalation of metal oxide after ultrasonic treatment [S Mathialagin et al]. The comparison between the XRD pattern of ZnO-TiO_2-GO composite in international journals and my observed sample are shown in table 1.

Table 1. The XRD patterns of ZnO-TiO_2-GO composite in international journals

No.	Author and Journal name	XRD pattern
1	Mathialagin et al, jornal of Scientific.	
2	Guru Nisha Narayanan et al, I.Journals Chem Tech.	
3	Václav Štengl et al, Chemistry Central Journal.	
4	C.Pragathiswaranet al, Journal of applicable Chemistry	

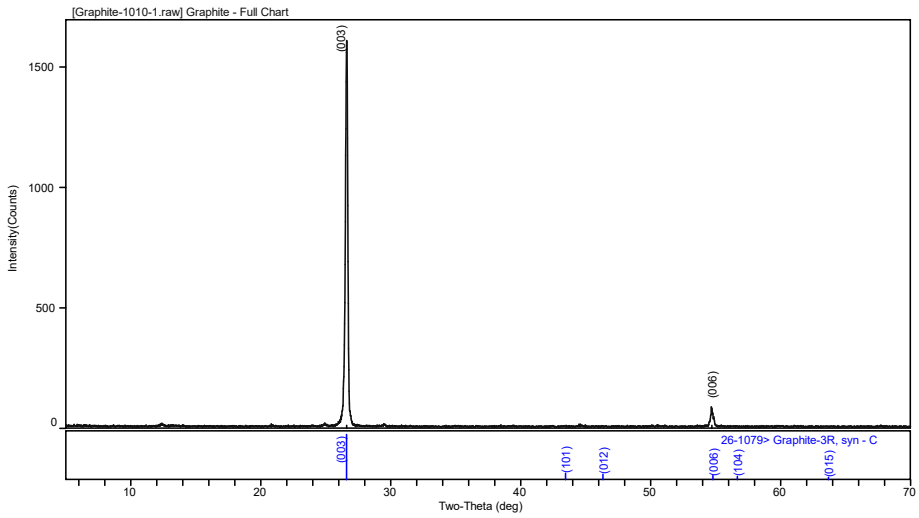


Figure 10. XRD pattern of graphite

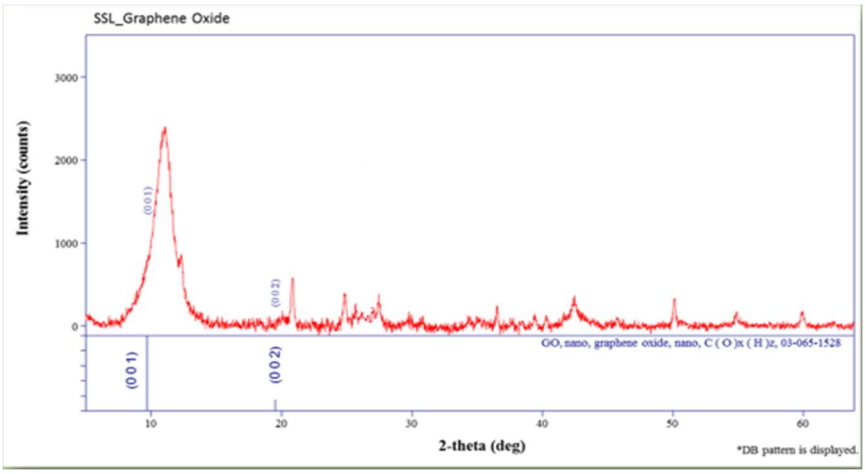


Figure 11. XRD pattern of graphene oxide

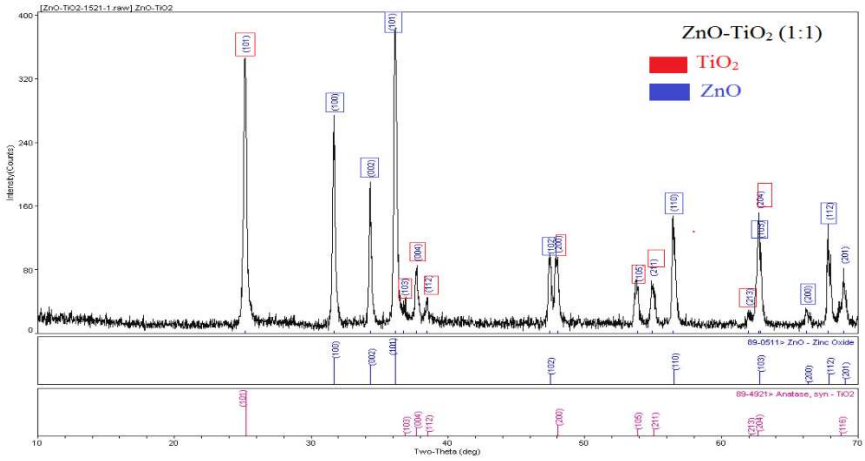


Figure 12. XRD pattern of ZnO-TiO_2 nanocomposite at 400 ˚C

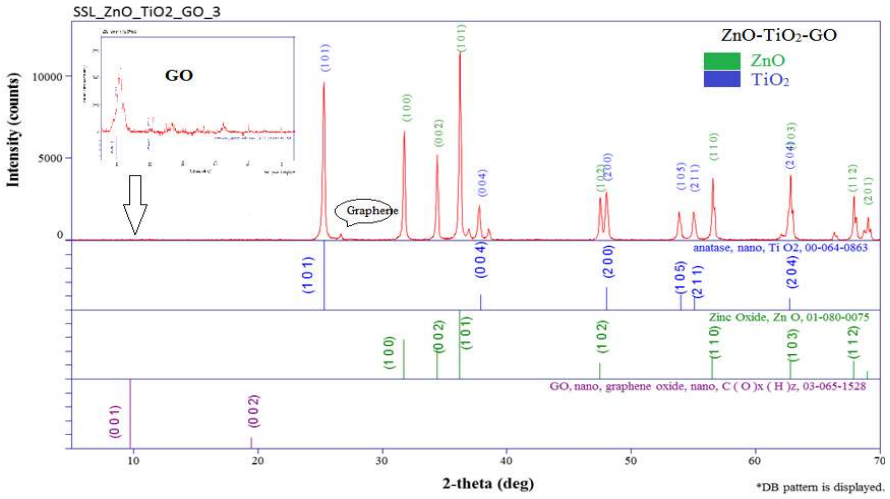
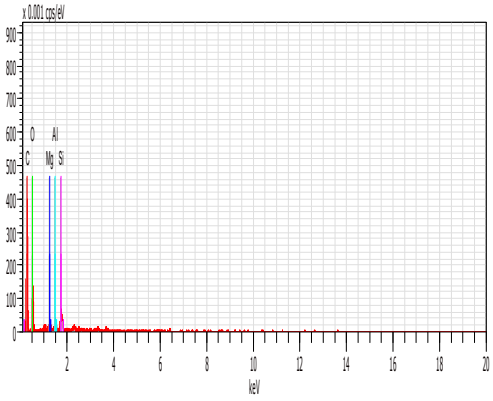


Figure 13. XRD pattern of ZnO-TiO_2-GO nanocomposite at 80 ˚C.

EDX analysis

The EDX figure of graphene oxide and the results of the EDX elemental for C and O elements of GO are describe in figure 14. The content of C is 67.35 % and the content of O is 29.75%. The mass ratio of C/O is 2.26. The international result of EDX for graphene oxide list in table 2. The EDX spectrum and elemental composition of ZnO-TiO_2 nanocomposite and ZnO-TiO_2-GO nanocomposite are also being described in figure15 and 16by EDX analysis.

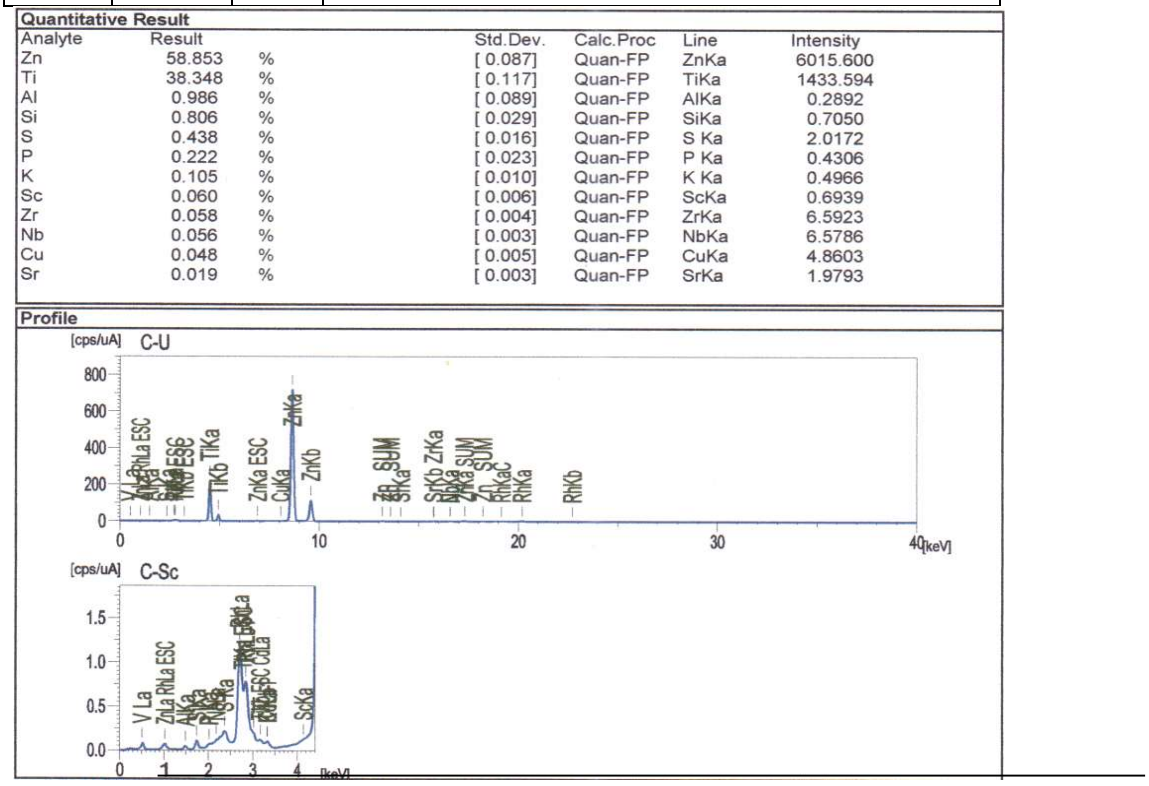


El	AN	Series	unn. C [wt.%]	norm. C [Wt.%]	Atom. C [at.%]	Error [wt. %]
C	6	K-series	67.35	67.35	74.03	13.5
O	8	K-series	29.75	29.75	24.55	8.2
Mg	12	K-series	0.53	0.53	0.29	0.1
Al	13	K-series	0.71	0.71	0.35	0.1
Si	14	K-series	1.67	1.67	0.79	0.2
Total:			100.00	100.00	100.00	

Figure14. EDX spectrum and elemental microanalyses for C and O elements graphene oxide

Table 3. The EDX elemental microanalyses for C and O elements in international journals

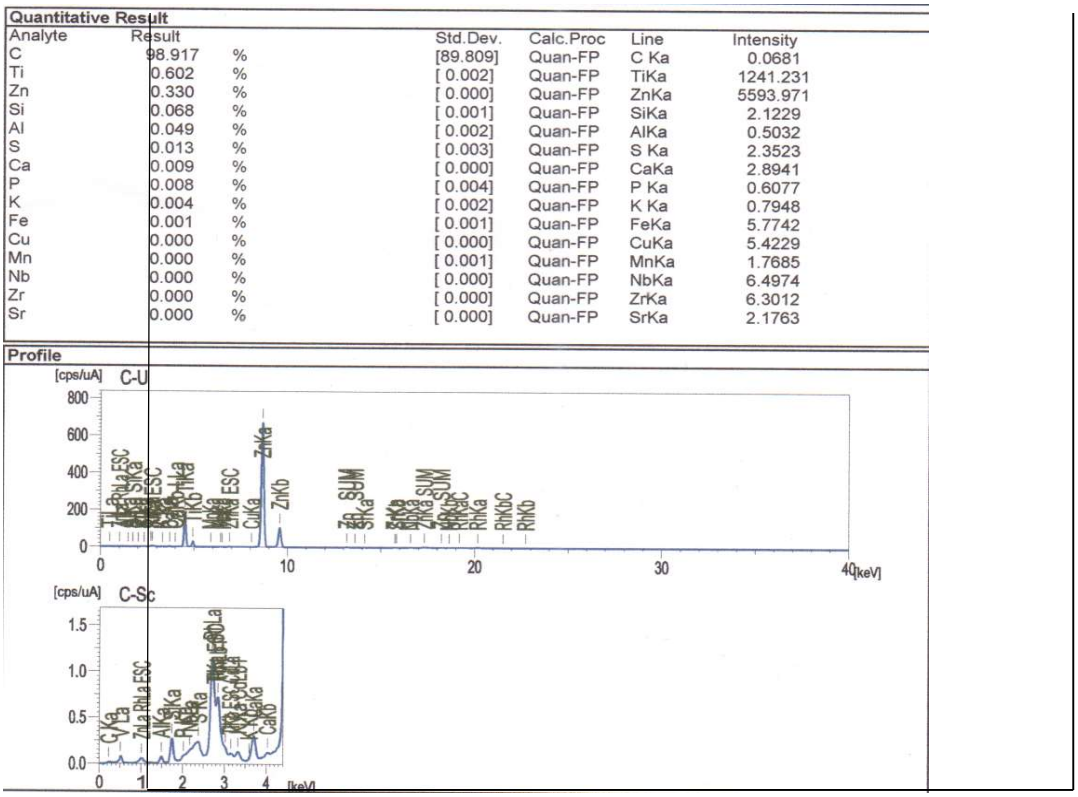
Element (wt. %)		**C/O**	**International Journals**
C	O		
51.32	44.27	1.15	Won-Chun Oh et al 2010 Journal of the Korean Physical Society, Vol. 56, No. 4, pp. 1097~1102
65.47	35.53	1.84	YunxianPiao et al 2011 The Royal Society of Chemistry
67.35	29.75	2.26	Observed sample



Quantitative Result

Analyte	Result		Std.Dev.	Calc.Proc	Line	Intensity
Zn	58.853	%	[0.087]	Quan-FP	ZnKa	6015.600
Ti	38.348	%	[0.117]	Quan-FP	TiKa	1433.594
Al	0.986	%	[0.089]	Quan-FP	AlKa	0.2892
Si	0.806	%	[0.029]	Quan-FP	SiKa	0.7050
S	0.438	%	[0.016]	Quan-FP	S Ka	2.0172
P	0.222	%	[0.023]	Quan-FP	P Ka	0.4306
K	0.105	%	[0.010]	Quan-FP	K Ka	0.4966
Sc	0.060	%	[0.006]	Quan-FP	ScKa	0.6939
Zr	0.058	%	[0.004]	Quan-FP	ZrKa	6.5923
Nb	0.056	%	[0.003]	Quan-FP	NbKa	6.5786
Cu	0.048	%	[0.005]	Quan-FP	CuKa	4.8603
Sr	0.019	%	[0.003]	Quan-FP	SrKa	1.9793

Figure 15. EDX spectrum and elemental composition of ZnO-TiO_2 nanocomposite



Quantitative Result

Analyte	Result		Std.Dev.	Calc.Proc	Line	Intensity
C	98.917	%	[89.809]	Quan-FP	C Ka	0.0681
Ti	0.602	%	[0.002]	Quan-FP	TiKa	1241.231
Zn	0.330	%	[0.000]	Quan-FP	ZnKa	5593.971
Si	0.068	%	[0.001]	Quan-FP	SiKa	2.1229
Al	0.049	%	[0.002]	Quan-FP	AlKa	0.5032
S	0.013	%	[0.003]	Quan-FP	S Ka	2.3523
Ca	0.009	%	[0.000]	Quan-FP	CaKa	2.8941
P	0.008	%	[0.004]	Quan-FP	P Ka	0.6077
K	0.004	%	[0.002]	Quan-FP	K Ka	0.7948
Fe	0.001	%	[0.001]	Quan-FP	FeKa	5.7742
Cu	0.000	%	[0.000]	Quan-FP	CuKa	5.4229
Mn	0.000	%	[0.001]	Quan-FP	MnKa	1.7685
Nb	0.000	%	[0.000]	Quan-FP	NbKa	6.4974
Zr	0.000	%	[0.000]	Quan-FP	ZrKa	6.3012
Sr	0.000	%	[0.000]	Quan-FP	SrKa	2.1763

Figure 16. EDX spectrum and elemental composition of ZnO-TiO_2-GO nanocomposite

FTIR analysis of Graphene Oxide

Infrared absorption spectrum diagram (FTIR) of graphene oxide was shown in figure 17. FTIR analysis allows qualitative discussion of the sturucture of graphene oxide. According to the FTIR analysis the peak at 1082.10 cm^{-1} and 1384.94 cm^{-1} which are attributed to the C-O bond, confirming the presence of oxide functional groups after the oxidation process. The peak at 1626.05 cm^{-1} can be attributed to the stretching vibration of C=C bond. The 3408.33 cm^{-1} correspond to O-H group of water molecules absorbed on graphene oxide. The presence of these oxygen containing groups reveals that the graphite has been oxidized.

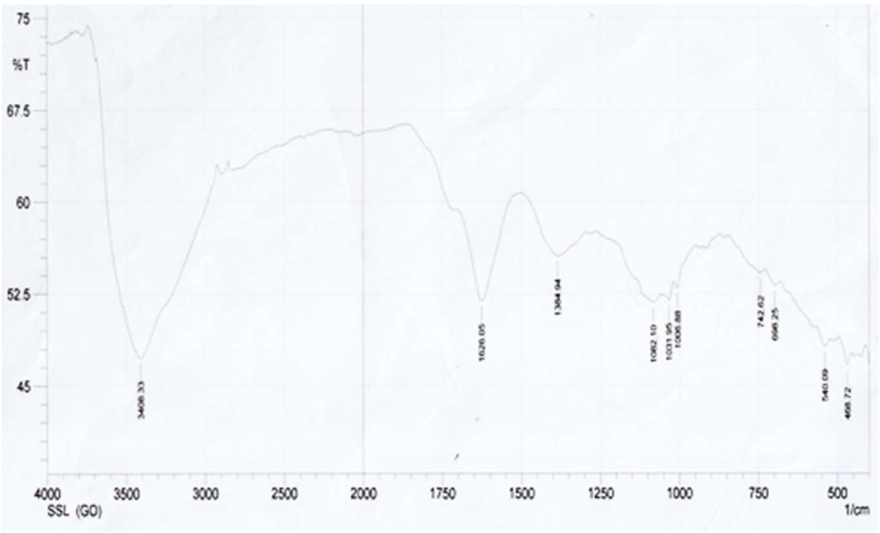


Figure 17. FTIR spectrum of graphene oxide

UV-Visible analysis

The UV-Vis spectra of GO, ZnO-TiO_2 nanocomposite and ZnO-TiO_2-GO nanocomposite powders were recorded with respect to the glass substrate placed in the reference beam using beam spectrometer in the range 190 to 1100 nm. The absorption spectrum of GO, ZnO-TiO_2, ZnO-TiO_2-GO deposited on glass substrate were shown in figure 18, 19 and 20. The GO shows high absorption coefficient in the UV region (290 nm). [In aqueous suspension, measurements reveal strong optical absorption in the UV (Mark Lundie et al)]. UV-Vis spectra of ZnO-TiO_2 nanocomposite exhibited at 289 nm and 372 nm and ZnO-TiO_2-GO was 543 nm and 645 nm respectively. The optical band energy (E_g) of the semiconductor is calculated from the relation.

$$\alpha h\upsilon = A (h\upsilon - E_g)^n \quad \text{——————} (1)$$

where α is the absorption coefficient, A is a constant (independent from υ), n is the exponent that depends upon the quantum selection rules for the particular material, h is the planck's constant and E_g is the energy band gap. A plot of $(\alpha h\upsilon)^2$ versus $h\upsilon$ shows intermediate linear region, the extrapolation of the linear part can be used to calculated the E_g from intersect with $h\upsilon$ axis as shown in figure 21, 22 and 23. The resultant values of E_g for pure GO is found

to be about 3.93 eV and Zn-TiO_2 nanocomposite is 3.8 eV. The value of E_g for ZnO-TiO_2-GO nanocomposite is found to be 4.083 eV.

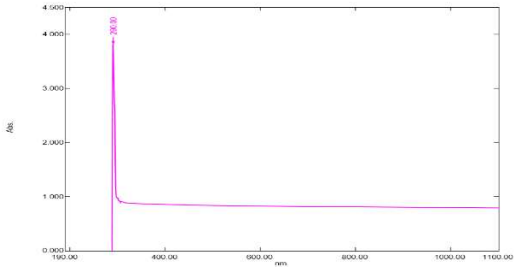


Figure 18. The absorption spectrum of graphene oxide

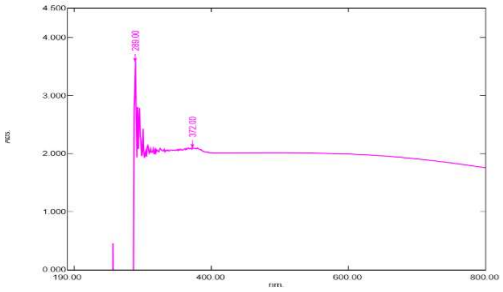


Figure 19

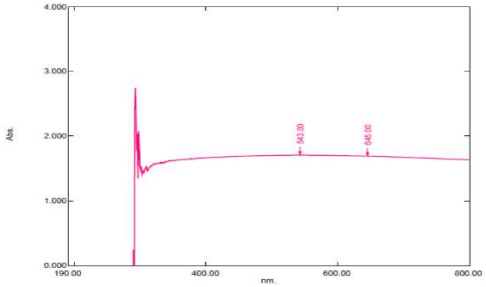


Figure 20

The absorption spectrum of (Fig.19) ZnO-TiO_2, (Fig.20) ZnO-TiO_2-GO nanocomposites

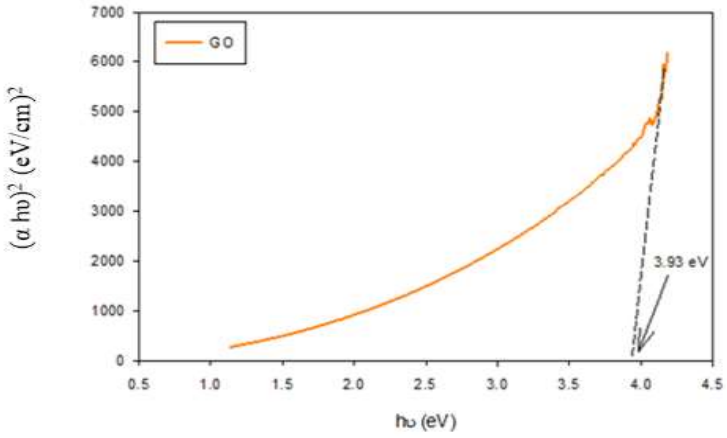


Figure 21. Plot of $(\alpha h\upsilon)^2$ vs photon energy ($h\upsilon$) for graphene oxide

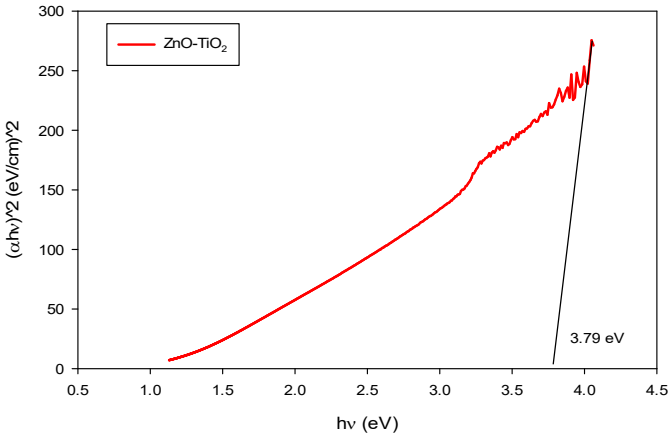


Figure 22. Plot of $(\alpha h\upsilon)^2$ vs. photon energy ($h\upsilon$) for ZnO-TiO_2 nanocomposite

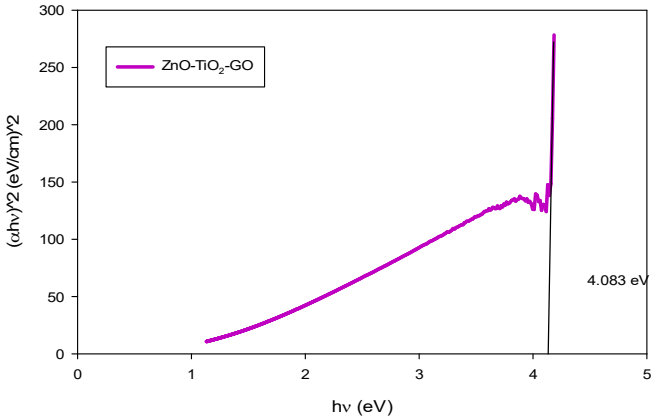


Figure 23. Plot of $(\alpha h\upsilon)^2$ vs. photon energy ($h\upsilon$) for ZnO-TiO_2-GO nanocomposit

AFM analysis

The AFM figures of topography, the amplitude, the 3D image and line profile of ZnO-TiO_2 and ZnO-TiO_2-GOnanocomposite are shown in figure 24 and 25. The particles sizes of ZnO-TiO_2 nanocomposites are estimated from particles height to avoid tip-convolution effects. According to the amplitude line profile most of the particles are agglomerate and the average particles sizes of ZnO-TiO_2 nanocomposite are about 78 nm. In ZnO-TiO_2-

GO nanocomposite, it is observed that ZnO-TiO_2 nanocomposite are well attacted onto the surface of GO sheet. The average particles sizes are also estimated form the particles height and the average particles sizes are about 30 nm- 50 nm.

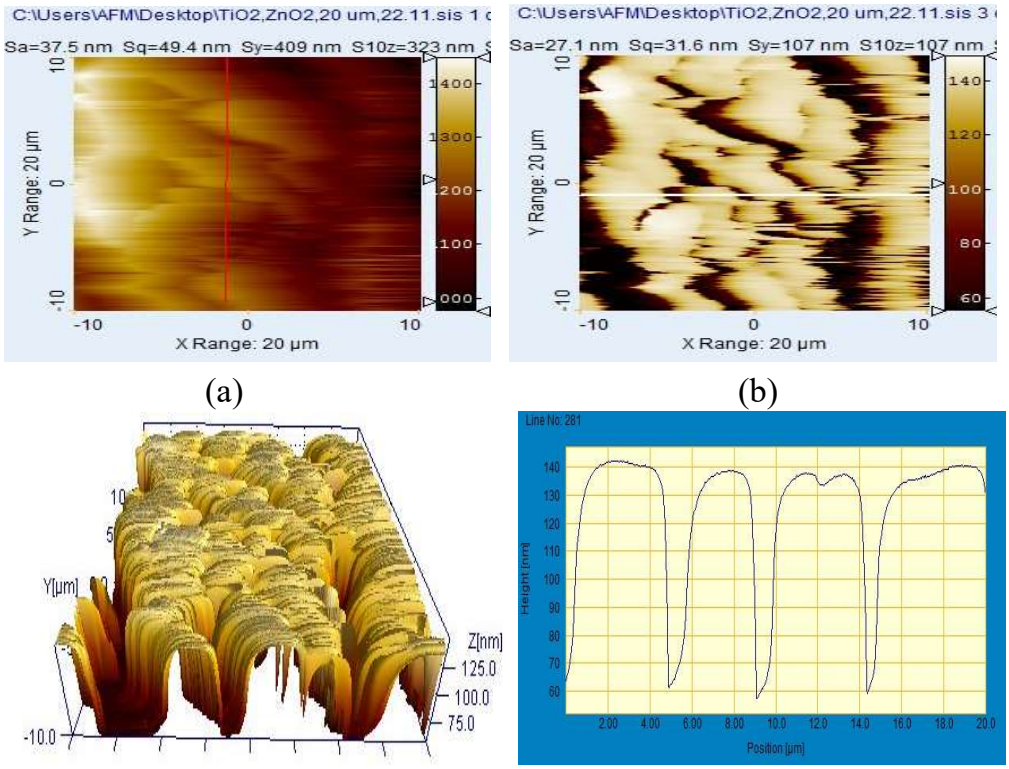
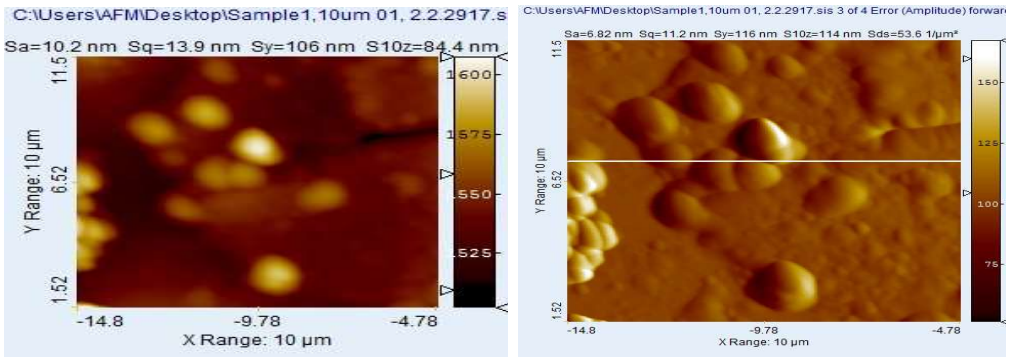
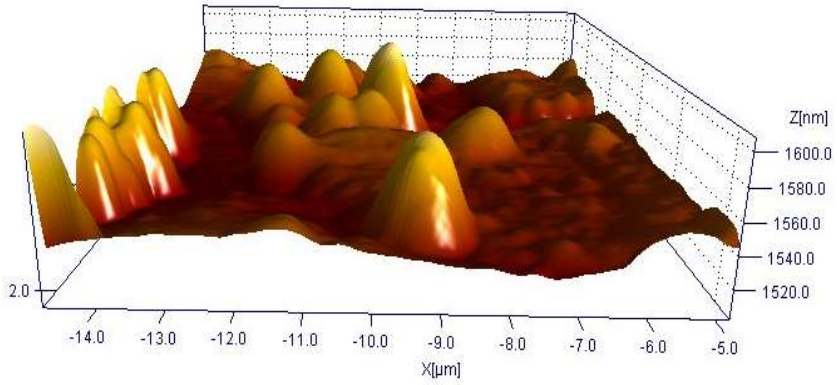
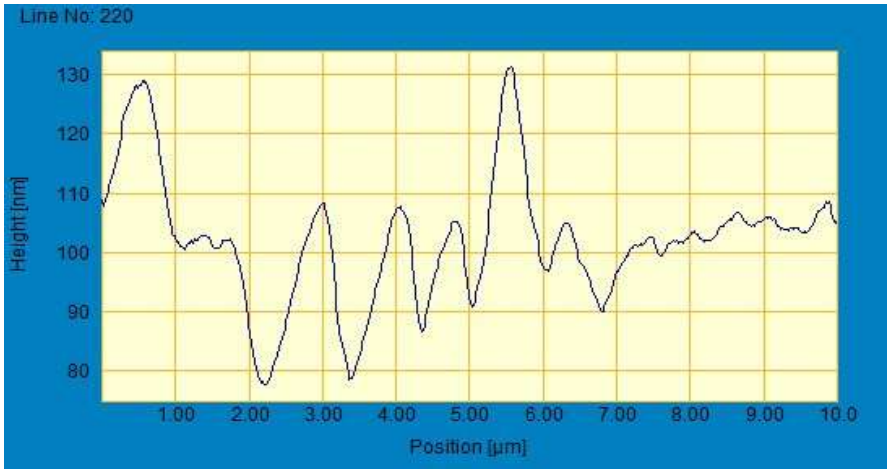


Figure 24. The topography (a), the amplitude (b), the 3D image (c) and line profile for ZnO-TiO_2nanocomposites (d)





(c)



(d)

Figure 25. Thetopography (a), the amplitude (b), the 3D image (c) and line profile for ZnO-TiO_2-GO nanocomposites (d)

FESEM analysis

The surface structure was estimated again by the field emission scanning electron microscope (FESEM). The FESEM images of GO, ZnO-TiO_2 and ZnO-TiO_2-GO nanocomposite was shown in figure 26, 27 and 28. In figure 26, surface of the graphene oxide was rough with some crumpling and agglomeration, which may be attributed to the residual oxygen containing

functional groups (e.g. -OH). According to the FESEM image of ZnO-TiO-$_2$nanocomposite (figure 27), the nanocomposite particles are also agglomerated and non-uniformed in size. Differentiation between ZnO and TiO_2 in the composites was not possible by FESEM owing to the similar electron density of Zn and Ti. The FESEM image of ZnO-TiO_2-GO nanocomposite was shown in figure 28and according to the SEM image, it is clearly seen that the GO sheets are decorated by ZnO-TiO_2 nanocomposite particles. The GO nanosheets act as a bridges for the connection between different ZnO-TiO_2 nanocomposite particles.

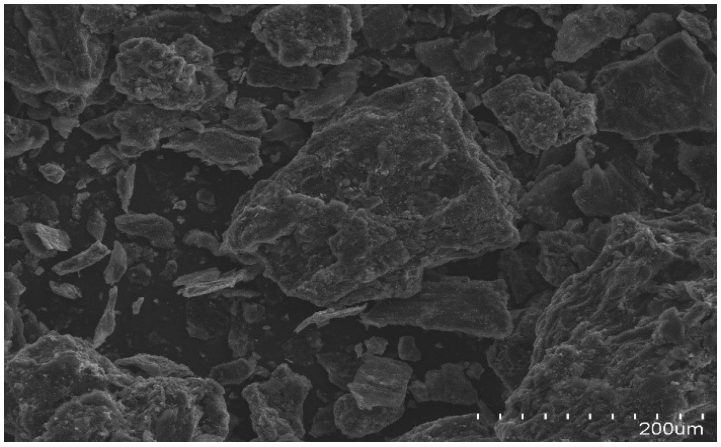
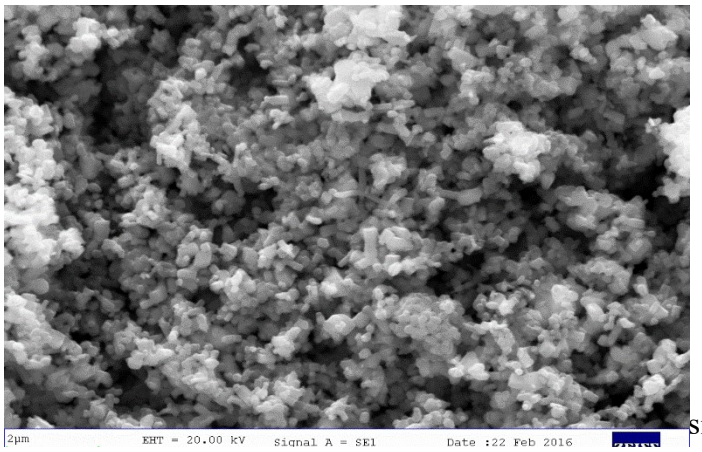


Figure 26. FESEM image of Graphene Oxide



site

Figure 27. FESEM image ofZnO-TiO_2 nanocomposite

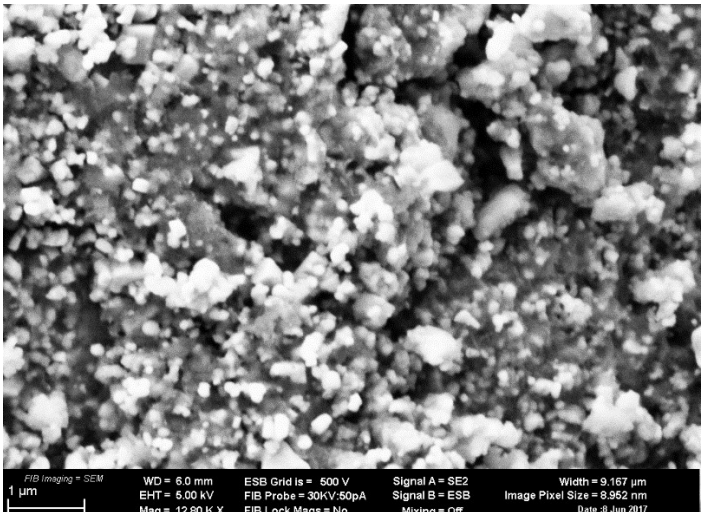


Figure 28. FESEM image of ZnO-TiO_2- GO nanocomposite

Conclusion

Graphene oxide (GO) was prepared by Hummer method. And then, ZnO-TiO_2 nanocomposite was prepared by simple mechanochemical activation method and the ZnO-TiO_2-GO nanocomposite was prepared by simple mechanical stirring followed by ultra-sonication. The XRD results of GO shows 2θ of 11.07° with the interplanar distance of 0.799 nm. Graphite, which showed a strong and sharp diffraction peak at 2θ=26.61° has the interplanar distance of 0.334 nm. The increase in interplanar distance of GO is due to the existence of oxygen functional groups. In ZnO-TiO_2-GO nanocomposite, the main diffraction peak of GO is absent and it probably lead to partial reduction of GO to graphene and a weak peak at 2θ = 26.50° appears. This might be due to the low amount and relatively low diffraction intensity of GO in comparison with the diffraction intensity of ZnO-TiO_2 nanocomposite. Moreover the other possibility is due to the intercalation of metal oxide after ultrasonic treatment. In EDX analysis, the content of C is 67.35 % and the content of O is 29.75% in GO. The mass ratio of C/O in GO is 2.26.According to the FTIR analysis, the –OH groups were be found and the presence of these oxygen containing groups reveals that the graphite has been oxidized. UV-Vis spectra of GO exhibited maximum absorption peak at 290 nm, ZnO-TiO_2 nanocomposite exhibited at 289 nm

and 372 nm and ZnO-TiO_2-GO was 543 nm and 645 nm respectively.The value of E_g for GO, $ZnTiO_2$ and ZnO-TiO_2-GO are found to be about 3.93 eV, 3.8 eV and 4.083 eV respectively.According to the AFM analysis, the particles in ZnO-TiO_2 nanocomposite areagglomerate and the average particles sizes are about 78 nm. In ZnO-TiO_2-GO nanocomposite, ZnO-TiO_2 nanocomposite are well attacted onto the surface of GO sheet and the average particles sizes are about 30 nm- 50 nm. In FESEM image, GO sheets are decorated by ZnO-TiO_2 nanocomposite particles. The GO nanosheets act as a bridge for the connection between different ZnO-TiO_2 nanocomposite particles and can be concluded it has the good behavior of nanocomposite.

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