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# Energy Band Gap Investigation of Polystyrene Copper Oxide Nanocomposites Bombarded with Laser



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ARTICLE INFO	ABSTRACT
Article history: Received 21 October 2019 Received in revised form 21 December 2019 Accepted 21 December 2019 Available online 4 March 2020	The presence of metal nanoparticles in polymer matrices modified their thermal, mechanical, electronic, optical and catalytic activity. Introduction of 2nd manipulation strategy, such as laser irradiation has enabled further tuning of the polymer-metal oxide nanocomposite (Polystyrene CuO Nanocomposite (PS CuO NCs)) properties. A low dose of laser irradiation to PS CuO NCs has enabled the investigation of the polymer molecular restructuring that depended on metal nanoparticle concentration and laser dosages. Copper oxide (CuO) nanoparticle was synthesized by the chemical reduction method and incorporated inside the polymer (Polystyrene) matrices. The 2nd manipulation strategy by using laser irradiation to these PS CuO NCs was later investigated through Ultraviolet-Visible (UV-Vis) and Fourier Transform Infrared (FTIR), Atomic Force Microscopy (AFM) and surface profiler in determining the energy band gaps of the bare PS and PS CuO NCs. The topography of the PS significantly changed after adding the copper oxide nanoparticles that filled the PS-interconnected pores with the spike. The decrease in energy band gaps of the PS molecules into carbonaceous materials which corresponded to laser dosages and metal nanoparticles concentration. The energy band gaps of the PS MNCs can be tuned from 4.29 eV (non-radiated) to 1.4-1.95 eV proves that the energy band gaps of PS MNCs can be modified using two manipulation factors, which are metal nanoparticles concentration and laser dosages. The energy gaps of the PS MNCs are able to be tuned from Insulator-Semiconductor-Conductor and the carbonaceous materials ranging from graphite, graphene to diamond-like carbon.
Metal nanoparticles; polystyrene; copper oxide absorption; band gap	Copyright © 2020 PENERBIT AKADEMIA BARU - All rights reserved

#### 1. Introduction

The basic principle of Photolithography is irradiating Electron or Ultra Violet beam to polymer resists materials, such as PMMA (Poly (methyl methacrylate)), or PS (Polystyrene) to change their

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solubility property of the irradiated polymers [1]. Two possibilities: The polymer resists molecular transform their structures to either become more crosslinking (binding up) (positive resist) or the second possibility is scissoring (breaking up) (negative resist) upon irradiation. Photolithography research is now shifted from pure patterning capabilities toward research on fabricating new materials through irradiation interaction with polymer molecules. Direct patterning of carbonaceous materials is found to reduce steps for fabricating electronic devices and could be used to develop an all-carbon based graphene field-effect transistor [2]. Incorporating or doping polymer resist materials with other materials, such as CNT (Carbon Nanotube) and nanoparticles are recently gaining interest in fabricating photonic and optical waveguide [3-6]. Nevertheless, problems arise in analyzing the chemical, physical and structure changes of the irradiated doped and un-doped polymer resist, as the exposure region could be less than 30 nm spot sizes, where analysis tools such as XRD (X-Ray Diffraction), FTIR (Fourier-transform infrared spectroscopy) or Raman Spectroscopy need to be incorporated as in-situ system with high precision scanning tools, such as FESEM (Field Emission Scanning Electron Microscope).

The inability to investigate irradiated PS when bombarded with electron beam irradiation was realized by Agam *et al.*, [7], where the interaction of the electron beams to shrink polystyrene nanospheres of 500 nm was difficult to analyze. In order to prove that the irradiated PS has changed its chemical property, investigation through reactive ion etching proved that the irradiated nanospheres polystyrene became denser and more resistant to the reactive ion etching process. It was found that the electron beam exposure of 9.9 mC/cm<sup>2</sup> dose at room temperature for a longer duration, decreased the diameter of the polystyrene spheres of 500 nm by  $\sim$ 13%. The shrinkage of the spheres offers a new route for nanospheres lithography where the gaps between the spheres can be accurately tuned. The change in the chemical properties of the irradiated PS was also found to have the same effect as irradiated by laser, where irradiated nanospheres PS are found to be resistant to Reactive Ion Etching (RIE) and organic solvents. Combining reactive ion etching process and organic solvent removal of unreacted/non-radiated polystyrene, fabricating of irradiated polystyrene nanodots in star-like patterns are possible and depending on the irradiation [8].

The difficulty of analyses the chemical, physical and structural changes of the irradiated PS nanospheres has led to the modification of the experimental design, where the type of irradiation sources, low power laser beam, long time exposure and larger exposure area are being introduced. These changes have led to the evolution of the research approach [9,10]. As low irradiation changes the polymer resist molecule slowly corresponding to the time of exposure and the large area of exposed region has enabled the investigations of the irradiated PS with many analysis tools, such as FTIR, XRD, Raman Spectroscopy and XPS.

It is found that the structural transformation of PS does not only correspond to laser irradiation but it is also enhanced by the presence of metal nanoparticles, that enables the tuning of the energy band gap of the PS metal nanocomposites by two factors: laser irradiation dosages (duration of exposure) and concentration of metal nanoparticles in PS matrices. In this report, the facile technique was used in tuning the energy band gap of Polystyrene Metal Composite (PS and Cu), which could be tuned from 1.4- 1.95 eV and the fabrication of graphene-graphite-diamond like carbonbased on laser exposure duration. The possibilities are huge including in this report, where the polystyrene metal composite can be used as water remediation material in absorbing heavy metal in synthetic water.



## 2. Polystyrene: Degradation and Manipulation

The polystyrene is a basic polymer built by two main elements, which are C, H and with two basic structures that consist of the backbone of C-C bonding and the benzene ring with repeating the formula of [11]. Photodegradation of PS has been investigated by many researchers to understand the ageing and stability of PS to sun light and other irradiation sources, especially toward UV radiation between maximum wavelength absorption,  $\lambda$ max=250 nm to longer wavelength of 300 nm [12-14]. The degradation of PS has also recently become an important factor in preserving the environment quality as PS are now becoming as pollutant to the environment due to PS is actively used in everyday materials, such as utensils and toys based materials [15].

The degradation of PS is also used in the photolithography technique, where polymer resist materials, such as PMMA and PS are irradiated and become either more soluble or resistant to organic solvent. Therefore, these changes are used in fabricating fine patterns on semiconductor substrates. Overexposure or longer duration of either electron beam, ultra-violet or laser irradiation to polymer resist materials, such as PMMA and PS is another research trend in the direct fabricating of carbonaceous materials [16]. It is found that overexposure of PMMA has fluctuated the chemical behavior of the irradiated materials, from soluble to non-soluble or becoming negative to positive type polymer resist materials. This chemical change is called zwitter characteristic phenomena [17-19].

Realizing that polymers degrade when exposed to radiation, it has the potential to be applied in creating cheap radioactive sensors. It is found that doping PVA (Polyvinyl alcohol) with crystal violet has increased the polymer-dope sensitivity, where the chemical structural changes of the dyed PVA films lead to the increase in their acidity that changes the color of the dyed PVA [19]. Investigations of longer exposure of gamma-ray to polystyrene by 30 kGy for 1-5 hours were also done to investigate the chemical, morphology and optical properties modification. Jibrin *et al.*, [20] found that the energy band gap of gamma-ray irradiated PS has decreased from 2.02 eV (bare PS) to 1.72 eV in 5 hours (longer) gamma-ray exposure.

The magnetic, mechanical and optical properties of the polystyrene were reported to improve when they were embedded with semiconductors or metal nanoparticles into the polymer matrix, besides being exposed to UV (ultra-violet) or ozone treatment [21-23]. The heat transfer or thermal conductivity of polystyrene can also be enhanced by the addition of metal nanoparticles. The potential of nanoparticles materials doped into polymers have shown interesting research trend in creating a polymer-metal composite with tunable magnetic, optical, chemical and electrical properties.

Degradation and manipulation of polystyrene can be realized through heat, chemical, doping with nanoparticles and laser irradiation which increased the potential not only as polymer resist materials, but also as wave absorption and coating materials [24]. These chemical changes, either by doping or laser irradiation will definitely change the energy band gap of the doped and irradiated polystyrene. In this research, Polystyrene Copper Oxide Nanocomposite (PS CuO NCs) with various doping ration of CuO were irradiated with low-intensity laser dosages; and it was found that the energy band gap has decreased which could be due to the doping concentration and laser dosages.

# 2.1 Energy Band Gap

The energy band gap for semiconductor materials can be investigated through the UV-Vis spectrum [25,26]. The UV-Vis data obtained from the transmittance characteristic of the PS CuO NCs



with various doping ratio and laser dosages were used to calculate the energy band gap by using Eq. (1) the value of optical absorption coefficient ( $\alpha$ ):

$$\alpha = \frac{1}{d} \ln\left(\frac{1}{T}\right) \tag{1}$$

where d is the thickness of the sample and T is the transmittance 24. Absorption coefficient ( $\alpha$ ) obeys the relation for the high photon energies (hv). Using Tauc's Eq. (2) the direct and indirect bands can estimate the energy band gap [27].

$$\alpha h v = B(h v - E_g)^n \tag{2}$$

where B is the band tailing parameter which relies on the electron-hole mobility, h is the energy of photon, is the sample band gap and n is constant that ascertain the type of transition, such as for the direct allowed is n = 1/2 and n = 3/2, for transitions of the forbidden n = 2 and for the indirect allowed and forbidden transitions respectively is n = 3.

# 3. Polystyrene: Degradation and Manipulation

# 3.1 Synthesis of Polystyrene

The synthesis of polystyrene particles used via nanoprecipitation technique [28-31]. Expanded Polystyrene (EPS) waste was obtained from packing material and heated to 150 °C in a hot air oven for 12 hours to remove any volatile matters that may agglutinate. The 20 mg expanded polystyrene was weighed and diluted in tetrahydrofuran (THF) of 4 mL with a ratio of 5mg/mL. The EPS/THF was mixed with 20 mL deionized water using magnetic stirrer and micropipette in a ratio of 1:10, to evaporate the THF the sample was loaded into an oven at 50 °C for 12 hours and finally achieved the end product needed.

# 3.2 Synthesis of Copper Oxide Nanoparticles

Copper sulphate pentahydrate (99.995% according to Merck company) (CuSO<sub>4</sub>.5H<sub>2</sub>O) with 0.01 M solution was prepared in the deionized water. Ascorbic acid solution with 0.02 M was prepared separately in the deionized water and (CuSO<sub>4</sub>·5H<sub>2</sub>O) was added to this ascorbic solution under continuous magnetic stirring. 1 M solution of NaOH in the deionized water was added in order to adjust the pH. After all the solution was stirred for 30 minutes at room temperature, (0.01 M) solution of NaBH<sub>2</sub>O was added during the continuous stirring. The reaction was completed after continuous stirring for 15 minutes in the ambient atmosphere. Copper is spontaneously oxidized by atmospheric oxygen and becomes copper oxide nanoparticles [32]. then copper oxide was mixed with polystyrene with different rates, the composites were mixed using vortex mixture for 10 minutes.

## 3.3 Laser Exposure

The schematic diagram of Figure 1 depicts the laser condition of Nd-Yag of 1064 nm with a frequency of 5Hz used to modify the PS CuO NCs structure. The PS CuO NCs with various concentrations are exposed to He-Ne laser with various time duration.





**Fig. 1.** Continuous laser Nd-Yag laser set-up in bombarding the samples to modify their chemical and energy gap property

# 4. Results and Discussions

4.1 Ultraviolet-Visible (UV-VIS) SHIMADZU Spectrophotometer

Figure 2(a) shows the absorption spectra of CuO at 252.50 nm was obtained from the chemical reduction method as indicated in black. Figure 2(a) the red line is the 257.00 nm and broad absorption at 594.50 nm absorption spectra of PS. The absorbance of polystyrene is higher compared to the absorbance of copper oxide nanoparticles. The optical characterization of PS, CuO and PS/CuONPs was analyzed in order to identify the energy band gap of them before and after exposure to the Nd-YAG radiation.

The UV-Vis absorption spectra in Figure 2(b) shows the shifts of peak absorption spectra where all of the samples had been modified by doping with metal oxide nanoparticles and irradiated with different times taken. The 1:2 ratios of PS/CuO nanoparticles in Figure 2(b) shows (black color) that the sample for 0 min at peak 263.50 nm shifted to the 260.00 nm and 250.50 nm as the time of exposure was increased to 5 min (red color) and 10 min (blue color) at the blue shift. The absorbance decreased as the time of the sample being exposed to Nd-YAG irradiation increased. The ratio 1: 4 composites, shows that the peak also shifted due to the time bombardment which 250.50 nm was the peak 0 min shifted to the 258.00 nm and 263.00 nm at the blue shift (not shown). The ratio of doped metal oxides nanoparticles and the time exposed Nd-YAG irradiation affected the properties of Surface Plasmon resonance samples, and that caused the changes in the resonant wavelength which corresponds to the blue shift or redshift.



**Fig. 2.** Raw UV-Vis Spectra data for (a) polystyrene and copper oxide nanoparticles, (b) polystyrene-copper oxide nanoparticles (1:2) at various dosage laser



# 4.2 Energy Band Gap

The values of the energy band gap at different ratio concentrations and time of exposure were determined by the linear line of plots  $(\alpha hv)^2 = 0$  from the graph of  $(\alpha hv)^2$  versus hv as shown in Figure 3. The Tauc's Eq. (2) shows the relation of incident photon energy hv absorption coefficient  $\alpha$ . The extrapolations of the straight lines give the direct optical band gap where the value transition, n use in this research is n = 2 (direct transition). decrease of the energy band gap of composite, the increased concentration may be due to the increase in the structural disorder of the polymer films with increased ratio metal oxide nanoparticles. The increase of time exposure Nd-Yag radiation in Figure 4 shows that the optical energy band gap has decreased. The decreased value of optical energy band gap is due to the enhanced diffusion rate in the forbidden energy level of  $\pi$ -electron of the polymer formation of defects or carbonaceous clusters created. The values of the energy band gap are summarized in Table 1.





Tabla 1





Fig. 4. Energy band gap of (a) PS:CuO 1:2 (0 min), (b) PS:CuO 1:2 (5 min), and (c) PS:CuO 1:2 (10 min)

The value optical energy band gap		
Samples	Energy band gap (eV)	
PS	2.00	
CuO	3.97	
PS: CuO (1:2) for 0 min	4.29	
PS: CuO (1:2) for 5 min	1.95	
PS: CuO (1:2) for 10 min	1.52	
PS:CuO (1:4) for 0 min	2.68	
PS: CuO (1:4) for 5 min	1.88	
PS: CuO (1:4) for 10 min	1.38	

## 4.3 Fourier Transform Infrared (FTIR)

The IR characteristic absorption peaks of PS in Figure 5(a) were observed at 3026.13 c correspond to the stretch of =C-H because of the aromatic rings. The peak at 2921.70 c attributed to the asymmetrical and symmetrical stretching vibration of the C. The peaks at 1492.65 and 1461.58 attributed to the stretching vibration of bond C=C on the benzene ring. The peak at 696.51 assigned to the bending vibrations C-H that out of the plane of the benzene ring. The IR characteristic absorption of CuO was observed at 3337.36 attributed to the vibration of hydrogen-bonded O-H stretch assigned to the presence of the hydroxide group. The peaks at 1592.07 correspond to the Cu-O symmetrical and asymmetrical stretching. The peaks range from 631.80 until 967.38 due to the C-H out of the plane deformation and the peak 1331.06 observed the metal-oxygen bond.

The irradiated samples are affected compared to the non-irradiated samples which show the structural changes as the result of the Nd-YAG radiation shown in Figure 3(b). The FTIR spectrum indicated that the intensity of bands 3307.88cm-1 for the sample PS/CuO ratio 1:2 non-irradiated shifted to the 3307.80cm-1 and 3307.57cm-1 due to the irradiation. The intensity of the ratio 1:4 observed in Figure 3(c) shows that 3307.46cm-1 also shifted to 3308.02cm-1 and 3307.75cm-1 after irradiation. The intensity of bands ratio 1:2 at 1635.15cm-1 shifted to 1635.23cm^-1 for 5 min and 1635.22cm-1 for 10 min. The ratio of 1:4 also shows the band at 1634.93cm-1 shifted to 1635.31cm-1 for 5 min and 1635.46cm-1 for 10 min of irradiation.





**Fig. 5.** FTIR spectra of (a) polystyrene and copper oxide nanoparticles, (b) PS/CuONPs (1:2) at various time bombardment and (c) PS/CuONPs (1:4) at various time bombardment

## 4.4 Atomic Force Microscopy (AFM)

Figure 6 shows the materials could have copper oxide nanoparticles as the spike can be seen clearly, where for the polystyrene usually their image can be seen as smooth and the existence of pores as in Figure 6(a) taken from AFM analysis. The topography of the PS significantly changed after adding the copper oxide nanoparticles that filled the PS-interconnected pores with the spike. The nanoparticles of CuO could be exposed more to the surface of topography ratio 1:2 (b) 5 min irradiated compared to the (c) for 0 min. The laser irradiation changes the structure of composite but would have to be confirmed by another technique, such as TEM. Figure 6(a) and 6(b) for the polystyrene-copper oxide nanoparticles (1:4) also shows that the topography for 6(a) is smoother as there are polystyrenes on the surface, compared to the 6(b) where there are spikes on the surface after being irradiated with laser.





**Fig. 6.** Atomic force microscopy images obtained from polystyrene at various time exposure (a) topography of non-irradiated PS, (b) topography of CuONPs

## 5. Conclusions

Polystyrene and polystyrene-copper oxide nanoparticles prepared were successfully treated by the Nd-YAG irradiation with different concentrations of copper oxide nanoparticles (1:2 and 1:4) and time bombardment (0 and 5 min). In this research, the exposure of PS/CuONPs by the Nd-YAG irradiation has systematically changed the chemical, optical and morphology of the samples. The chemical resistance of the PS by the Nd-YAG irradiation shows the carbonaceous material formed based on the chemical properties by FTIR. The energy band gap obtained by Tauc equation proved that the energy band gap decreased with the increasing (from 4.29 eV to 1.38 eV)of time bombardment and the concentration of the copper oxide nanoparticles.

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