

FULL PAPER

# Solar-driven Degradation of 2-Chlorophenol Using PANI/GO as Photocatalyst

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## Abstract:

Confiscation of organic pollutants from the environment is one the most important topics recently. The contamination of our water body that comes from several pollutants lead to several serious effects to the environment. The development of green and economical method in reducing the contamination is well discussed. The main aim of this research is to characterize and investigate the solar driven photocatalytic degradation of 2-chlorophenol (2-CP) from aqueous solution with the aid of polyaniline (PANI) incorporated graphene oxide (GO) composite (PANI/GO). PANI was incorporated with GO via *in-situ* oxidation polymerization method using ammonium persulfate (APS) as oxidizing agent to form composite. Three compositions of PANI/GO composites were synthesized with the ratio of PANI and GO of 1:1, 1:4 and 9:1. PANI, GO and PANI/GO composites were characterized comprehensively using Fourier transform infrared (FTIR), UV-visible (UV-VIS) and XRD spectroscopy analysis. Morphology was analyzed by capturing the images using Scanning electron microscope (SEM) with Energy Dispersive X-ray (EDX) analysis setup. The characterization studies revealed that, the PANI/GO composites have been successfully synthesized. Degradation of 2-CP was conducted using PANI/GO composites under solar irradiation for 3 hours. The degradation results disclosed that, PANI/GO composite with composition ratio of 1:1 possessed the highest degradation percentage with 75 % at degradation time of 150 minutes. Therefore, PANI/GO composites are one of the ideal catalysts in order to degrade 2-CP.

**Keywords:** polyaniline; graphene oxide; photocatalytic degradation; 2-chlorophenol; solar irradiation

## 1. Introduction

Global environment continually have been contaminated by various type of pollutants exist at different location [1]. The European Union (EU) classified 132 hazardous chemicals should be controlled in water based on their stability, bioaccumulation and toxicity [2]. Among these well-known organic pollutant, phenolic group attracted attention in the field of photocatalytic degradation due to its prominent production in the water around the world. Phenolic compound such as 2-chlorophenol (2-CP) is a common pollutant in water that has high toxicity to human being even at low concentration exposure [3]. 2-CP commonly used in agricultural sector as one of the compounds in the fertilizer. The exposure

to 2-CP may cause skin irritation, gastrointestinal problems and in some cases pose a serious ecological risk as environmental contamination [4].

The US Environment Protection Agency (USEPA) enforces very strict regulation for 2-CP amount in wastewater where the threshold value must be lower than 1 ppm. Therefore, finding an economical and practical technique to degrade 2-CP from the environment is crucial. Several methods such as adsorption, distillation and membrane extraction have been implemented for the degradation of various pollutants from the environment [5]. Unfortunately, these methods do not comprehensively eliminate the pollutant and the detrimental sludge still remains after the treatment [6]. Recently, photocatalytic

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degradation becomes one of the most efficient techniques to degrade pollutants and has been considered as a green, convenient and promising technology in water treatment [7]. This technique utilizes the light source and forms electron-hole which produce radicals that can convert contaminant to safe and less poisonous substances. Special feature of photocatalytic degradation is easily degrade the organic and biological pollutants which possesses high photochemical stability by effective utilization of solar light irradiation [8].

Heterogeneous photocatalyst containing hybrid materials of conducting polymers and semiconductor composites have been widely utilized in various fields for instance charge storage materials, catalysts, solar cells and electronics. Heterogeneous photocatalyst needs a relatively mild system to generate reactive free radicals that able to remove the toxic and refractory nature of organic contaminants [9]. Therefore, the integration of semiconductor with conducting polymers such as polyaniline (PANI) can give a synergistic effect between catalyst and polymer, eventually decrease the energy of band gap to make it available in the visible light [10].

Graphene oxide (GO) is one of the semiconductor that contains graphene layer with carbonyl, epoxide and hydroxide that makes them available for functionalization [11]. GO can act as electron acceptor or donor and can cooperate with many free radical species formed during photocatalytic degradation because it contains numerous delocalized electrons.

On the other hand, PANI is a well-known  $\pi$ -conjugated p-type conducting polymers with many interesting characteristics such as narrow band gap, high absorption coefficient in the visible light range and good environmental stability that can provide great potential application in the photocatalytic activity [13]. Therefore, combination of PANI and GO is expected to improve thermal, structural, electrical, optical properties towards various aspects in photocatalytic applications [14].

The aim of this research is to characterize and investigate PANI/GO composite as a photocatalyst to degrade 2-CP under solar irradiation. To the best our knowledge, no report was found on the application to degrade 2-CP by

using the heterogenous PANI/GO composites. Hence, a detailed analysis was done to optimize the best PANI to GO ratio that will work excellent in photocatalytic degradation applications.

## 2. Results and Discussion

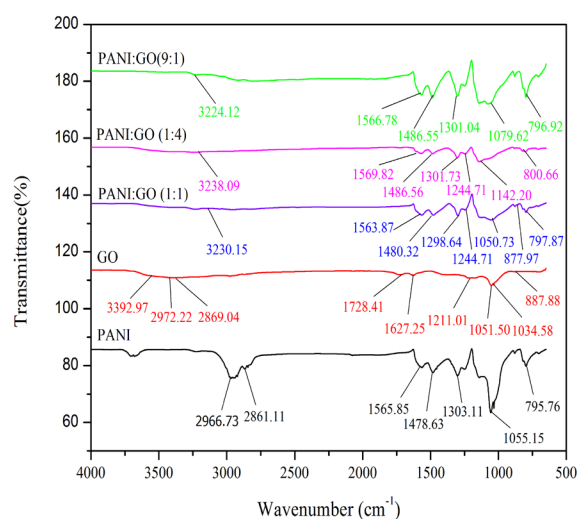
### FTIR analysis

Figure 1 shows various peaks in the spectrum on PANI, GO and PANI/GO composites, within the range of 650 – 4000  $\text{cm}^{-1}$ . The characteristic peak of PANI can be confirmed by a strong peak at 2966.73 and 2861.11  $\text{cm}^{-1}$  represented to C-H stretching. The present of C=C stretching vibration band at 1565.85 and 1478.63  $\text{cm}^{-1}$  assigned to the structures of benzenoid and quinoid ring, respectively. Besides, there is also stretching vibration of C-N that was observed at 1303.11  $\text{cm}^{-1}$ . The strong absorption at 1055.15  $\text{cm}^{-1}$  was the characteristic peak of doped PANI, corresponding to the in-plane bending vibration of C-H caused by the protonation [17]. The bending peaks located at 796.76  $\text{cm}^{-1}$  can be allocated as a band of benzene ring thus, it was a mono-substituted benzene ring [18,19].

The graphene oxide showed the broad peak at 3392.97  $\text{cm}^{-1}$  assigned to the hydroxyl (-OH) groups stretching vibration at GO surface. Then, the peaks at 2972.22 and 2869.04  $\text{cm}^{-1}$  were attributed to the stretching vibration of  $\text{sp}^2$  hybridized carbon. Another peak represented; GO was at 1728.41  $\text{cm}^{-1}$  which assigned to C=O stretching of carboxylic acid (-COOH) groups. The stretching vibration at 1211.01 and 1051.50  $\text{cm}^{-1}$  were assigned to C-O-C (epoxy) and stretching of C-O, respectively. The peaks at 1627.25 and 887.88  $\text{cm}^{-1}$  were corresponding to C=C bond which is aromatic groups [20].

Many functional groups of GO were shifted to higher/lower bands or disappeared within the PANI/GO composites due to the increase mass of the compound. However, in most cases, the increasing ratio of the GO shifted the peaks to the higher wavenumber. Meanwhile, in terms of peak intensity, the peaks at  $\sim 1500 \text{ cm}^{-1}$  and  $\sim 1400 \text{ cm}^{-1}$  showed increasing intensity as the amount of GO increased in the PANI matrix [20]. The stretching vibration at 1728.41  $\text{cm}^{-1}$  in GO is cannot be observed in all PANI/GO composites because the reduction of GO took place due to polymerization of aniline clearly indicated the

presence of more of imine units than amine units in the PANI chains [21]. In addition, appearance of C–N peak stretching at  $\sim 1300\text{ cm}^{-1}$  was in good agreement to the existence of covalent bonding of PANI on GO through reaction with epoxide ring existed on the basal plane of GO [20]. All characteristic peaks of PANI and GO were detected in the PANI/GO composites.



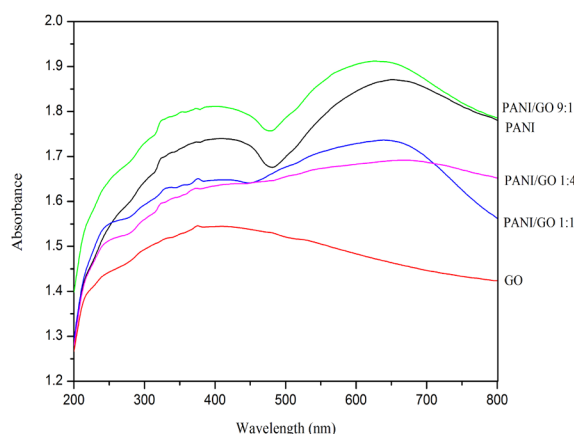
**Figure 1.** FTIR spectrum of PANI, GO, PANI/GO 1:1, PANI/GO 1:4 and PANI/GO 9:1.

### UV-Vis Spectroscopy Analysis

Figure 2 shows the UV-Vis spectra of pure PANI, GO and PANI/GO composites within the wavelength range of 200–800 nm. The three absorption peaks were observed at  $\sim 324$ ,  $\sim 410$  and  $\sim 641$  nm which attributed to  $\pi$  to  $\pi^*$  and  $\pi^*$  to polaron peaks and  $\pi$  to polaron peaks transitions of PANI, respectively. In addition, for the characteristic absorption peaks of GO was displayed at 238 nm due to  $\pi$  to  $\pi^*$  transition of aromatic C=C bonds. The C=O bond in GO can be detected around 370 nm as a shoulder peak due to  $n$ - $\pi^*$  transition. The PANI/GO composites exhibited all the characteristic absorption peak of PANI and GO [22]. The pattern peak of PANI/GO 1:1 and PANI/GO 9:1 were almost the same with the absorption peak of pure PANI. Conversely, the design of PANI/GO 1:4 peak that contains more GO was likely to the absorption peak of pure GO.

The resulting peak showed that the addition of GO into the PANI matrix shifted the absorbance ratio. The presence of pink, blue and

green shifts with variation in the quantity of peaks in PANI/GO composites was due to charge transfer occurring between PANI and GO because of doping of carboxylic acid of GO to PANI backbone [23]. Obviously, the intensity of pure PANI peak was the highest, suggesting that photogenerated electrons and holes in large extent. The intensity of the absorbance ratio slightly decrease when incorporated with the GO indicating that the addition of PANI and GO effectively inhibited the recombination of photogenerated charge with holes [24]. However, PANI/GO with the ratio 9:1 had the highest intensity due to the excess of PANI in the GO that facilitated the photogenerated electrons and holes in large extent than the pure PANI where it was obviously not an ideal photocatalyst.

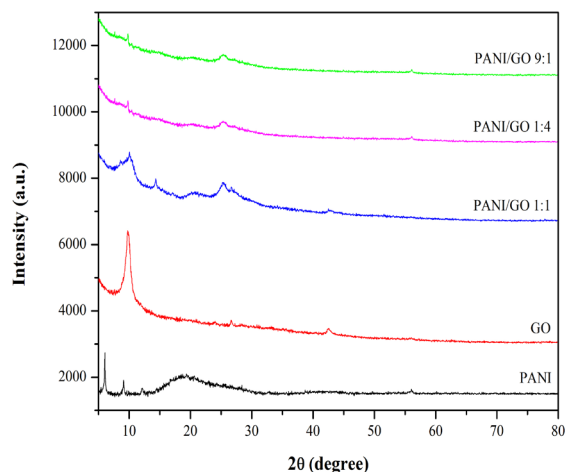


**Figure 2.** UV-Vis spectra of PANI, GO, PANI/GO 1:1, PANI/GO 1:4 and PANI/GO 9:1

### XRD Analysis

Figure 3 indicates diffractograms of PANI, GO and PANI/GO 1:1, PANI/GO 1:4 and PANI/GO 9:1 composites. The broad peak of PANI was observed at  $2\theta = 10$ – $30^\circ$  that represent the emeraldine PANI salts. Diffractogram of PANI showed low crystallinity due to the presence of conjugated benzenoid or quinoid chain in PANI [25]. GO diffractogram indicates the presence of intense peak at  $2\theta = 8.7^\circ$  that correspond to the interplanar space of GO sheet (001). Meanwhile, the XRD peak of all PANI/GO composites were found at  $2\theta = 8.7^\circ$  which indicated that the presence of GO in the compound, yet the degree of crystallinity and intensity decrease, as compared to the GO. This can be due to the intercalation of PANI chains between GO sheets

and the increasing of interspacing distance between GO sheets because of the addition of polymer matrix [26]. All the respective peak of PANI and GO present in the composite. Hence, in this study it had been confirmed that PANI/GO have been successfully integrated in the heterogenous composite system.



**Figure 3.** XRD diffractograms of PANI, GO, PANI/GO 1:1, PANI/GO 1:4 and PANI/GO 9:1.

### Morphology analysis

A comprehensive morphological analysis was conducted using SEM images on the synthesized PANI, GO and PANI/GO composites. Figure 4a showed the surface morphology of PANI was rod-like particle with some granular agglomeration. A small quantity of nanofiber also appeared in the PANI. Meanwhile, in GO the structure as in Figure 4b appeared layer-like structure, flaky structure and agglomerated. In Figure 4c it is clearly exposes that, GO sheets are homogeneously surrounded with PANI [27]. From the images, it revealed that the PANI has been successfully incorporated with GO.

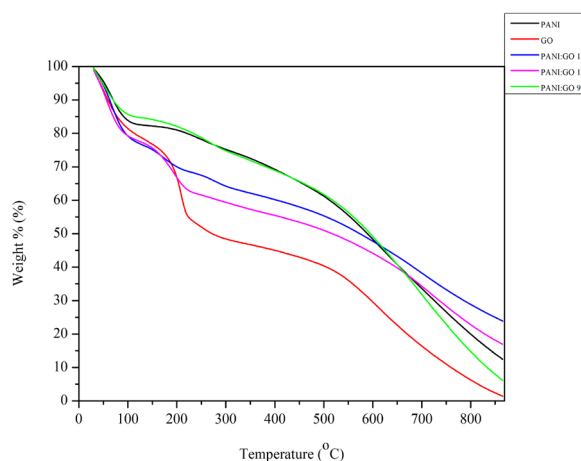
### Electron dispersive X-ray (EDX) analysis

Figure 4 also illustrated the EDX analysis of chemical composition (d) pure PANI, (e) GO and (f) PANI/GO 1:1 composite. Figure 4(d) showed the chemical compositions that appeared in PANI which was carbon (C) and nitrogen (N) as the characteristic peaks. Signals of Sulphur (S)

and Chloride (Cl) indicate that the PANI has been successfully doped by the APS and HCl. It can be seen higher value of C element in PANI than other which is 74.76%. Figure 4(e) showed a significant signals of carbon (C) and oxygen (O) was attributed to GO with the percentage composition of 71.80% and 28.20%, respectively. Figure 4(f) indicated the successful incorporation of GO within PANI matrix as the elements from both samples co-exist in the EDX spectra. PANI/GO composites exhibited similar EDX spectra as pure PANI and GO as discussed earlier. Therefore, the mapping analysis confirms the distribution of elements throughout the PANI matrix [10].

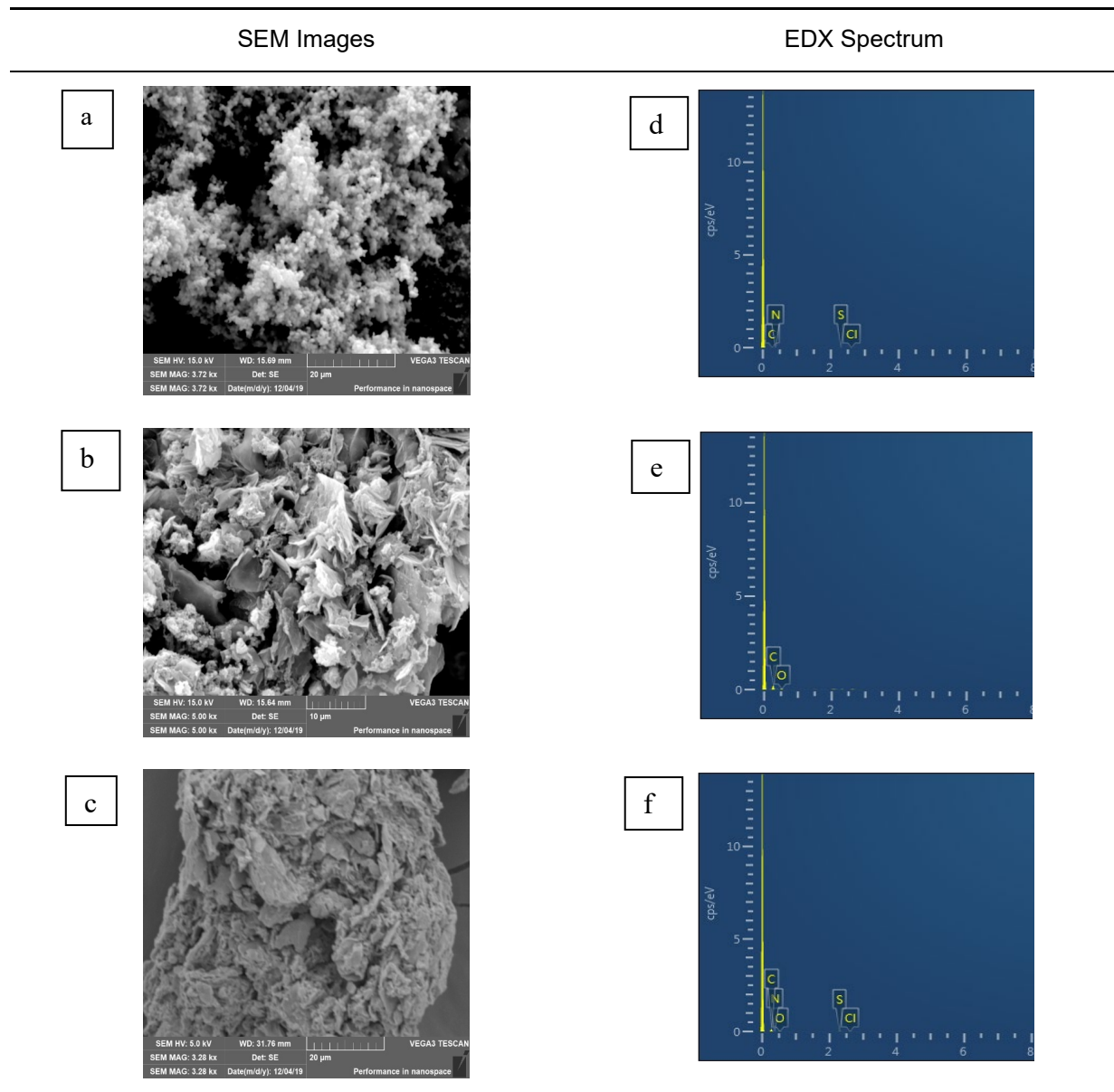
### TGA Analysis

The decomposition pattern of the samples was studied using TGA. As shown in Figure 5, major weight loss of GO was observed from 150 °C due to the pyrolysis of labile oxygen containing groups [28]. At 300 and 650°C, a slower mass loss of more stable oxygen functionalities can be observed. PANI loss of water molecules and low molecular weight, oligomers can be observed around 150°C and the degradation of polymer chains occurred right after 350°C [29]. Meanwhile, when the concentration of GO was increased, more oxygenated groups of GO were absorbed into the PANI matrix and during heating, the oxygenated groups of GO removed quickly resulting in lowering the thermal stability of PANI [23].



**Figure 5.** TGA analysis of pure PANI, GO and PANI/GO composites





**Figure 4.** SEM image of (a) PANI, (b) GO and (c) PANI/GO 1:1 with its respective EDX spectra of (d) PANI, (e) GO, (f) PANI/GO 1:1.

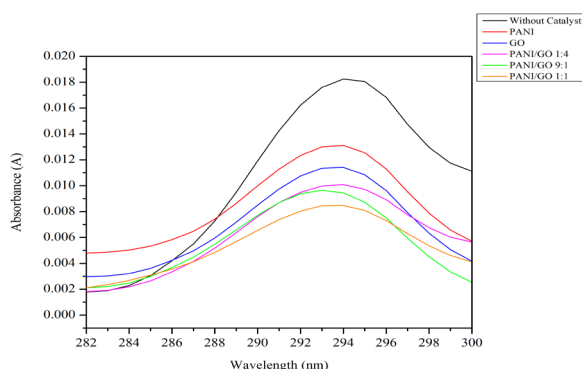
### Photocatalytic Activity Studies

#### *The comparison of photocatalytic activity of PANI, GO and PANI/GO composite*

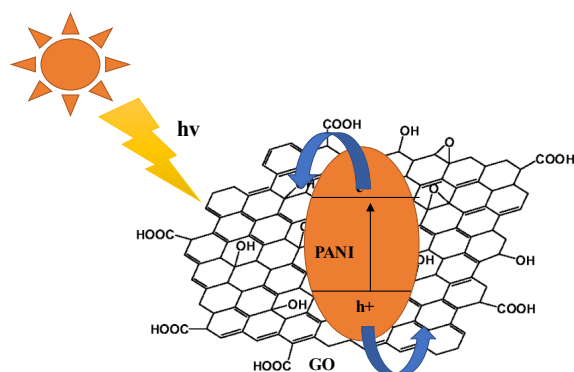
Figure 6 showed the degradation of 2-chlorophenol when performed without catalyst and with different catalyst which were PANI, GO and PANI/GO composites with different ratios. PANI and GO exhibited poor photocatalytic activity under visible light with only 30.28% and 39.72% percentage degradation, respectively. PANI and GO exhibited poor percentage of degradation due to recombination of electron/holes that leads to ineffective production

of radicals to degrade the 2-chlorophenols [12]. However, the photocatalytic activity for PANI/GO composite was much higher than PANI and GO. The degradation percentage for PANI/GO 1:1, PANI/GO 1:4 and PANI/GO 9:1 were 72.22%, 45.94% and 51.56%, respectively in 3 hours. The higher percentage degradation of PANI/GO composites indicates that heterojunction photocatalyst improved the catalytic activity. When, PANI integrated with GO, it helps efficient charge separation, tends to suppress the recombination rate of the generated electron-hole pairs. Under visible light irradiation, PANI could be excited to generate electrons and holes.

Figure 7 illustrates the photogenerated electrons in the conduction band (CB) of PANI would migrate to GO while the holes in the valence band (VB) of PANI could transfer to GO. As a result, the recombination of formed electrons and holes could be prevented greatly [13].



**Figure 6.** Different absorbance for different catalyst (condition: 0.01 g composites, 20 mL of 10 ppm of 2-chlorophenol and exposed to sunlight for 3 hours).



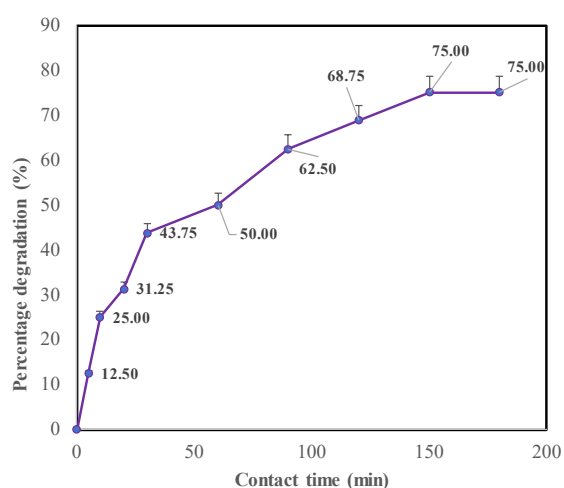
**Figure 7.** Schematic diagram of photogenerated electrons/hole in the PANI/GO composites

### ***The effect of contact time towards photocatalytic degradation of 2-CP***

PANI/GO 1:1 composite exhibited the highest percentage degradation; therefore the effect contact time were further carried out using 0.01 g PANI/GO 1:1 composite. The composite was exposed to sunlight and observed for every 30 minutes interval in 3 hours. The result of percentage degradation was presented in Figure 8.

Based on Figure 8 it can be observed that the percentage of degradation was increased as the exposure time to sunlight increased. This is

because of extra photon energy were absorbed by photocatalyst which provided by the sunlight. Thus, it will result in the formation of more hydroxyl radical that is significant for photocatalytic activity [8]. Furthermore, the results revealed that the percentages of degradation continuously increase until 150 minutes and the percentage remain constant until 180 minutes. Therefore, the maximum percentage degradation of 2-chlorophenol is 75% because when the time over than 180 minutes the photocatalyst become saturated in solution, thus it will lower the degradation efficiency [22].



**Figure 8.** Percentage degradation of 2-CP by using PANI/GO photocatalyst with 1:1 ratio.

## **3. Material and Methods**

### **Materials**

Aniline monomer, ammonium persulfate (APS), 2-chlorophenol (2-CP), graphite powder, sodium nitrate ( $\text{NaNO}_3$ ), hydrogen peroxide ( $\text{H}_2\text{O}_2$ ), potassium permanganate ( $\text{KMnO}_4$ ), ethanol, sulfuric acid ( $\text{H}_2\text{SO}_4$ ) and hydrochloric acid (HCl) were purchased from Sigma Aldrich and all the chemicals were analytical grade.

### **Preparation of PANI**

The PANI was synthesized by chemical oxidation method at  $0^\circ\text{C}$  for 24 hours. Initially, 15 mmol (1.4g) of aniline was dispersed in 1 M HCl followed by the dropwise addition of precooled APS for 1 hour with continuous stirring. The

mixture was left to stir for 24 hours at 0-5 °C to allow polymerization process to complete. The product obtained was filtered and washed with deionized water for several times until the filtrate became colourless. Lastly, the product was dried in an oven at 50 °C for the drying process prior to characterization and application.

### Preparation of GO

The GO was prepared by using modified Hummer's method. 1 g of graphite powder, 0.5 g of NaNO<sub>3</sub> and 23 mL of 18 M of H<sub>2</sub>SO<sub>4</sub> were added into Erlenmeyer flask and were stirred at 300 rpm for 1 hour at 0-5 °C. Then, 3 g of KMnO<sub>4</sub> was poured slowly and stirred vigorously for 1 hour. The mixture was removed from the ice bath and continued stirred at 300 rpm for 4 hours. The mixture was put into water bath until the temperature increase to 35 °C and continuously stirred at 300 rpm for another half an hour. The 250 ml of deionized water was added into the mixture and heated until the temperature raised up to 70 °C. The unreacted compound was removed by mixing of 30 % H<sub>2</sub>O<sub>2</sub> solution. The product was washed with 5 % HCl and deionized water. Finally, GO was centrifuged several times at 300 rpm for 10 minutes and dried in the oven at 70 °C for 12 hours.

### Preparation of PANI/GO composites

PANI/GO composites were prepared with three different compositions via in situ chemical oxidative polymerization. PANI and GO ratios were varied on the PANI/GO composites. Table 1 shows the formulation of each ratios. GO was dispersed by sonication for 30 minutes with 50 mL of HCl. Then, 15 mmol aniline was poured into the mixture and stirred at 300 rpm for 1 hour. 5 ml of ethanol was added to avoid aggregation. Then, in separate flask 15 mmol of APS and 1 M HCl were mixed. The pre-cooled APS mixture was added dropwise into the GO-aniline mixture for 1 hour with continuous stirring. The mixture was left to stir at 300 rpm for 24 hours at 0-5 °C in an ice bath to allow polymerization process. Upon the completion of polymerization, the composites were washed and filtered with water and ethanol repetitively and recovered by Buchner funnel filtration until the filtrate became colourless. The product was dried in oven for 12

hours at 60 °C [15]. The experiment was analyzed with different ratio in order to investigate the optimum ratio of PANI/GO composites in photocatalytic degradation application.

**Table 1.** PANI/GO composites formulation.

Ratios (PANI/ GO)	Aniline (mmol)	GO (mmol)	APS (mmol)
1:1	15	15	15
1:4	15	60	15
9:1	135	15	15

### Characterizations of PANI, GO and PANI/GO composites

PANI, GO and PANI/GO composites were characterized using attenuated total reflectance (ATR) - Fourier transform infrared (FTIR) between 600 cm<sup>-1</sup> to 4000 cm<sup>-1</sup> (Model 100 Spectrometer, Perkin Elmer). Next, UV-visible spectroscopy (model Lambda 750, PerkinElmer) was used to determine the absorbance of composites in the wavelength range of 200 – 800 nm. The crystallographic structure was determined by using Empyrean X-ray diffractometer (Model TESCAN VEGA3) by keeping 2θ = 10°-90° Cu, Kα radiations (λ = 1.5418 Å) at a scan of 0.02 sec<sup>-1</sup>. The surface morphology and elemental analysis was observed in scanning electron microscopy (SEM) spectrometry (Brand: TESCAN; Model: VEGA3) coupled with electron dispersive X-ray spectroscopy (EDX) Model Oxford Instrument, 50mm<sup>2</sup> (SEM-EDX) after sputter coating with platinum.

### Photocatalytic activity studies

The photocatalytic properties of PANI, GO and PANI/GO composites were evaluated by employing 2-CP as a target degradation product. Photocatalyst was added into 20 ml of 10 ppm of 2-CP solution and the mixture was exposed to the sunlight for 180 minutes. All the mixture solution was filtered, and the clear supernatant solution was measured for its absorbance at 295 nm by using UV-vis spectroscopy. The percentage degradation efficiency was calculated by using Equation (1):

Percentage degradation efficiency, (%) =  $\frac{A_0 - A}{A_0} \times 100$ .....(Eq. 1)

where  $A_0$  is the initial absorption of 2-CP and A is the absorption of 2-CP at sampling time after the degradation process [16].

#### 4. Conclusions

In conclusion, pure PANI, GO and PANI/GO composites were successfully prepared. PANI/GO composites with 1:1 ratio was successfully applied effectively in the degradation of 2-CP. FTIR, UV-Vis, XRD and EDX analyses confirmed the incorporation of GO in the PANI matrix. Among the PANI, GO and PANI/GO samples, PANI/GO (1:1 ratio) exhibited the highest photodegradation with the maximum percentage degradation of 2-chlorophenol which is 75% within the equilibrium time of 150 minutes. Overall, it has been proven that GO can be added into PANI matrix to improve the photocatalytic degradation of 2-CP.

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