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Photocatalytic Degradation of Phenol over Carbon Nitrides Prepared by Urea and Melamine Precursors

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31

Abstract. Carbon nitride (CN) has been well-known as the photocatalyst with a favorable visible light absorption ability for environmental treatment, such as the degradation of organic pollutants. The use of different precursors would result in the different reaction pathways to form CN, which then could affect the properties and photocatalytic activity. In the present study, we used two different precursors, which were urea and melamine, to prepare the CN. The CN samples were characterized by diffuse reflectance ultraviolet-visible (DR UV-Vis), fluorescence, and Fourier transform infrared (FTIR) spectroscopies. The resulted CN products were confirmed to have different optical properties to each other. The CN prepared from urea has a lower absorption edge and higher bandgap energy than the CN prepared from melamine. The presence of C=O groups could be detected on the CN prepared by urea precursor which led to the lower emission intensity than the CN prepared from melamine precursor. The CN prepared by urea gave 1.9 times higher photocatalytic activity (21.30%) than the CN prepared by melamine (11.05%) for photocatalytic degradation of phenol. The presence of defects in the CN prepared by urea was proposed to contribute to the photocatalytic activity.

INTRODUCTION

Phenol is identified as one of the toxic organic pollutants and is commonly found in wastewater from plastic, household, and medical product industry. As this compound can be absorbed rapidly through the skin then cause organ damage in long-term accumulation, many techniques have been developed to remove phenol completely from the wastewater [1–4]. Since phenol includes as a non-biodegradable compound and has high stability in water, it needs strong reactive oxygen species to be degraded into non-dangerous compounds. Hence, the advanced oxidation processes (AOPs) obtain great attention as a technique involving an oxidation reaction that forms hydroxyl radicals ($\cdot\text{OH}$) for breaking the organic pollutants [1]. One of the AOPs that is considered to be environmentally friendly is the photocatalytic reaction [2]. It is known as a green technology since it only uses light source, open air, and photocatalyst.

In order to fully utilize sunlight which consists of more visible light than UV light, it is important to develop the visible-light active photocatalysts. Graphitic carbon nitride (CN) is widely recognized as visible-light active photocatalyst that able to absorb the light up to 460 nm [5]. This makes it appear as a yellow solid and has medium bandgap energy, implying that it is able to generate more photons as compared to metal photocatalyst. Based on previous research reports, CN has been successfully used as an effective photocatalyst for energy conversion and environmental treatment applications, such as hydrogen evolution [6, 7], reduction of CO_2 to hydrocarbon fractions [8], and degradation of organic pollutants under visible light irradiation [9, 10]. The good performance of the CN could be related to its high chemical and thermal stability as the photocatalyst. The good stability of the CN could be due to its strong chemical bonds between the carbon and nitrogen elements, which would depend on how it is synthesized [11].

Generally, CN is synthesized by thermal condensation of nitrogen-rich precursor. There are various types of N-rich precursors that have been used to synthesize CN, such as cyanamide, dicyanamide, melamine, and urea [12].

6

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Since cyanamide and its derivative are toxic, the use of less hazardous melamine and urea precursors shall be more explored. It was reported that the condensation process of melamine was started from the formation of melam structure, following the removal of ammonia gas, then rearrangement to form melem, and by the end of the process was the polymerization of melem structure [13]. Meanwhile, the reaction pathway when using urea precursor was involving the formation of a mixture of cyanic acid, biuret, cyanuric acid, ammeline, and melamine structures before melem polymerization process [14].

The different reaction pathways would result in different degrees of polymerization, which would affect the properties and photocatalytic activity of the synthesized CN. Therefore, studying the effect of precursor to prepare the CN would be significant to clarify the important parameters to obtain highly active CN photocatalysts. In this study, we studied the utilization of different precursors, which were urea and melamine, for the preparation of CN. The obtained CN photocatalysts were evaluated for photocatalytic degradation of phenol under visible light illumination. We demonstrated that selecting the correct precursor was crucial to obtain the CN photocatalyst with high activity.

MATERIALS AND METHODS

General

Urea ($\text{CO}(\text{NH}_2)_2$, 98%) and melamine ($\text{C}_3\text{H}_6\text{N}_6$, 99%) were purchased from Sigma-Aldrich. Phenol ($\text{C}_6\text{H}_5\text{OH}$, 99.5%) was purchased from Merck. All the chemicals used in the synthesis procedure were prepared without any purifications. The DR UV-Vis spectra were recorded by JASCO V-760 UV-Vis spectrophotometer by using barium sulfate (BaSO_4) as the reference with the scan range of 200–600 nm. All the FTIR spectra were measured by JASCO FT/IR-6800 spectrometer in the scan range of 400–4000 cm^{-1} , using Attenuated Total Reflection (ATR) technique with the air as the baseline. The excitation and emission spectra as fluorescence property were carried out in JASCO FP-8500 spectrofluorometer. Shimadzu CTO-10AS VP equipped with a PDA detector was used as the high-performance liquid chromatography (HPLC) for analyzing the concentration of phenol after photocatalytic reaction.

Preparation of CN Photocatalysts

Urea and melamine were used as the source of carbon and nitrogen for the preparation of CN. The urea or melamine precursor (25 g) was calcined at 550 °C for 4 hours under limited air atmosphere. The rate of heating process was set to 2.2 °C/min to reach the temperature of 550 °C. After the calcination, the yellow solid was ground to obtain fine powder before it was used as a photocatalyst. The CN sample obtained from urea precursor was labeled as CN_u , while that obtained from melamine precursor was labeled as CN_m .

Characterizations of CN Photocatalysts

The characterization of DR UV-Vis spectra was carried out by measuring barium sulfate (BaSO_4) first as the reference before the CN samples. Each CN sample (5 mg) was put in the solid holder with a circular quartz glass. The measurement parameter was set by the scan range of 200–600 nm with the scan speed of 100 nm/min. The data was recorded in reflectance percentage (%R) versus wavelength, which then the %R was converted to be Kubelka-Munk function values. For obtaining the Tauc plot, the wavelength as the x-axis of DR UV-Vis spectra was converted to be photon energy (hv). The y-axis of the Tauc plot was obtained from the half squared of multiplication of Kubelka-Munk function values and the photon energy. The bandgap values were obtained by dividing the intercept to slope values of the linear equation from the fitting curve line of the Tauc plot. For the characterization of FTIR spectra, the air was recorded as the baseline, and then the CN sample (2 mg) was measured with the scan range of 400–4000 cm^{-1} . For the measurement of fluorescence properties, the CN sample (5 mg) was put onto the quartz glass in the solid holder. The measurement was recorded in the scan range of 250–400 nm for the excitation spectra, and in the scan range of 390–800 nm for the emission spectra with very low sensitivity.

13 Photocatalytic Activity Test

The photocatalytic activity was evaluated for the photocatalytic degradation reaction of phenol at room temperature. The CN photocatalyst (0.05 gram) was dispersed in 50 mL of aqueous phenol (70 ppm) in 100 ml of a round bottom flask. Prior to the visible-light illumination, the mixture was stirred in a dark condition for 2 hours to reach the adsorption equilibrium. The mixture was then stirred under visible light irradiation (Dolan-Jenner Fiber-Lite MI-157, 150-Watt Halogen lamp) for 24 hours. After the reaction, the solution was taken and separated from the photocatalyst using a syringe filter. The remaining concentration of phenol was analyzed by using high-performance liquid chromatography on a Shimadzu CTO-10AS VP equipped with a PDA detector with a monitored wavelength of 272 nm. The percentage of phenol degradation was calculated by following Eq. (1):

$$\text{Percentage of degradation (\%)} = \frac{C_o - C}{C_o} \times 100 \quad (1)$$

where C_o and C were denoted to the initial concentration of phenol and concentration phenol after visible light irradiation for 24 hours, respectively.

RESULTS AND DISCUSSIONS

Properties of CN

The physical appearances of the CN obtained from urea and melamine precursor are shown in Fig. 1. The colour difference between CN_u and CN_m was very clear. The CN_u showed a light yellow colour, while the CN_m gave more intense yellow colour. The different colour showed the difference in the polymerization degree; the more intense colour could correspond to the more condensed or polymerized CN.

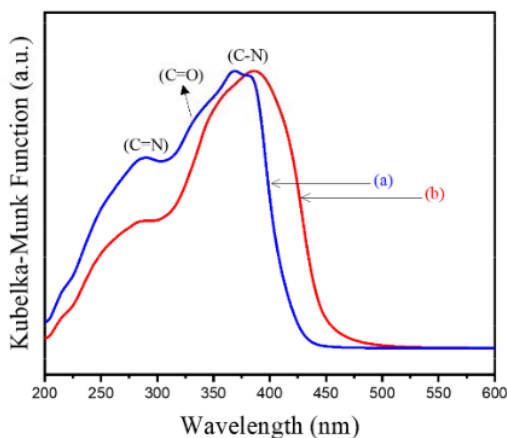


FIGURE 1. Colour appearances of (a) CN_u and (b) CN_m samples

In order to investigate the optical absorption properties of the CN samples, the DR UV-Vis spectra were measured and shown in Fig. 2. Both CN samples showed broad absorption peaks at UV and visible regions. As expected, the intensity of the light absorption in the visible region was higher on the CN_m than on the CN_u , supporting the more intense yellow colour in the CN_m . Moreover, the CN_u showed absorption peaks in the range of 200–430 nm, while the CN_m gave absorption peaks at longer wavelength range of 200–480 nm. The wavelength range of the light absorption on both CN samples was well-agreed with the appearance of the CN samples as yellow solid that can absorb the light up to the wavelength range of blue light [4, 15]. The CN_u sample gave absorption peaks at 287, 330, and 370 nm, which could be assigned to the $\pi \rightarrow \pi^*$ charge transfer at the $\text{C}=\text{N}$ groups in the 1,3,5-triazine unit, weak charge transfer at C=O group, and the $n \rightarrow \pi^*$ electronic transition at the terminal C-N

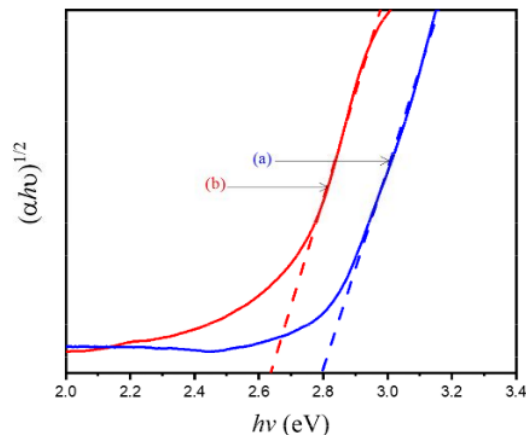
groups of the triazine aromatic ring, respectively [16–18]. As for the CN_m , similar absorption peaks could be detected at 292 and 387 nm, which were contributed from the $\pi \rightarrow \pi^*$ charge transfer at the C=N groups and the $n \rightarrow \pi^*$ electronic transition at the terminal C-N groups [16, 17].

The redshift to a longer wavelength was observed not only in the absorption edges but also the absorption peaks, when changing the urea precursor to melamine precursor. The red-shifting observed when using melamine precursor would be related to the higher degree of CN polymerization in the CN_m than in the CN_u , which would enhance the delocalization of electrons in the CN layer [19]. It was reported that the improved degree of polymerization could be affected by the length of the synthesis time [20]. Under the same synthesis time, the polymerization in the CN_u would be shorter than that in the CN_m . Urea should change to cyanic acid first and get through five steps until melamine was formed, which was then followed by the melamine polymerization, while melamine precursor merely would only need two steps to form the CN. Using melamine precursor allowed the melamine condensation to have enough reaction time for completing the polymerization. In contrast, the CN produced from urea precursor has uncondensed functional groups such as C=O groups at 330 nm [18]. This result confirmed that the CN_u has a lower degree of polymerization, which led to a narrower absorption edge.



11
FIGURE 2. DR UV-Vis spectra of (a) CN_u and (b) CN_m

Tauc plot was investigated to determine the bandgap energy of the CN_u and CN_m samples. As shown in Fig. 3, the CN_u has bandgap energy of 2.82 eV, while the CN_m has bandgap energy of 2.62 eV. The bandgap energy of CN_m was close to the reported value for mesoporous CN prepared by cyanamide, which was 2.6 eV [21]. The larger bandgap of the CN_u showed that the electron excitation in the CN_u shall occur at slightly higher energy than that of the CN_m . However, it was clear that both CN samples could absorb visible light irradiation of more than 400 nm, and thus, the electron excitations could occur on both samples under visible light irradiation.



2
FIGURE 3. Tauc plots of (a) CN_u and (b) CN_m .

12
 In order to identify the functional groups in the two CN samples, the FTIR spectra were measured. As displayed in Fig. 4, both CN_m and CN_u exhibited similar vibration bands at around 810 cm^{-1} and $1200\text{--}1640\text{ cm}^{-1}$ as the result of bonding interactions between carbon and nitrogen in the CN matrix [15, 16, 17, 21, 22]. The vibration band at 810 cm^{-1} was attributed to the bending modes of 1,3,5-triazine unit, whereas the bands at $1200\text{--}1640\text{ cm}^{-1}$ were contributed to the stretching mode of C–N heterocycles. The bands at 1206 cm^{-1} , and 1313 cm^{-1} corresponded to the medium and strong stretching mode of C–N, respectively, while the band at 1632 cm^{-1} was denoted to C=N stretching mode. In addition, the other vibration bands were observed at $3000\text{--}3300\text{ cm}^{-1}$ that indicated the presence of N–H bond from uncondensed amino groups in the form of NH_2 or NH, and the existence of O–H bond from the adsorbed water molecules at the surface of CN structure. The difference in the functional groups between the CN_u and the CN_m samples was the presence of a weak vibration band at 1680 cm^{-1} for CN_u . This vibration band corresponded to the C=O stretching mode in the CN_u structure [21, 22], in good agreement with the DR UV-Vis spectra discussed in Fig. 2. This result again confirmed that there was an incomplete condensation of CN when using oxygen-containing precursor such as urea.

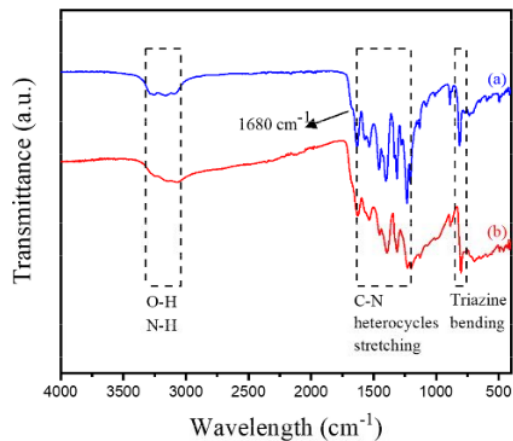


FIGURE 4. FTIR spectra of (a) CN_u and (b) CN_m

The fluorescence properties of the CN_u and CN_m were investigated by spectrofluorescence. Fig. 5 shows the excitation spectra of the CN samples. The CN_u gave similar excitation peaks to the CN_m , with two maximum intensities were observed at 277 and 369 nm. Analogy to their absorption peak from DR UV-Vis spectra, the excitation peak of 277 and 369 nm could be assigned to the $\pi \rightarrow \pi^*$ charge transfer at the C=N groups in the 1,3,5-triazine unit and the $n \rightarrow \pi^*$ electronic transition at the terminal C-N groups of the triazine aromatic ring, respectively [18, 21]. It was also clear that the excitation intensity of CN_m was slightly higher than the CN_u which well-matched with the better ability of the CN_m in absorbing the visible light.

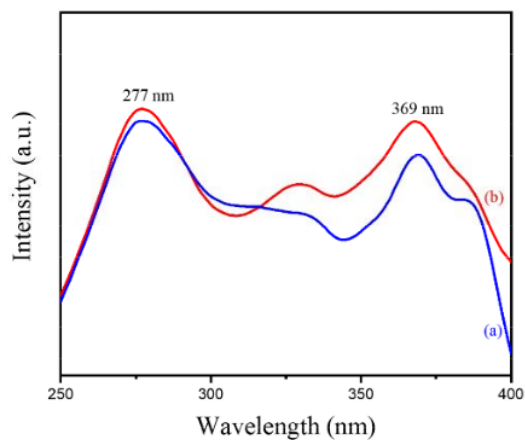
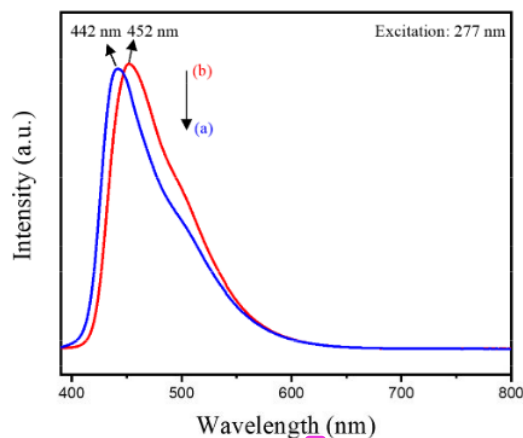
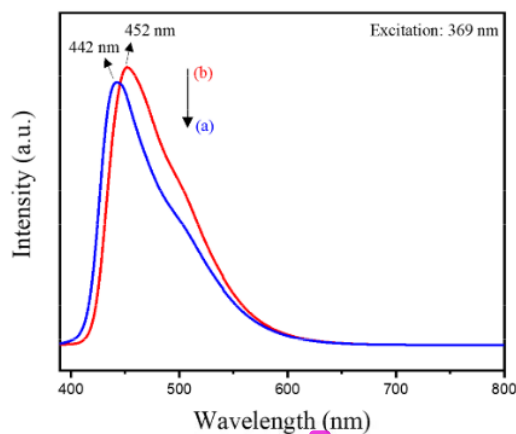


FIGURE 5. Excitation spectra of (a) CN_u and (b) CN_m monitored at the emission wavelength of 442 and 452 nm, respectively



5 **FIGURE 6.** The emission spectra of (a) CN_u and (b) CN_m monitored at the excitation wavelength of 277 nm



2 **FIGURE 7.** The emission spectra of (a) CN_u and (b) CN_m monitored at the excitation wavelength of 369 nm

2 Even though both CN_u and CN_m samples have excitation peaks at 277 and 369 nm, the emission peak on these two CN samples was different from each other. Either excited at 267 or 369 nm, the CN_m gave an emission peak at 442 nm, while the CN_u gave an emission peak at 452 nm. The emission spectra of these CN samples are shown in Fig. 6 and Fig. 7. Similar to the results from DR UV-Vis spectra, the emission spectra of CN_m were also shifted to the longer wavelength than the CN_u sample. In addition, the CN_u exhibited a slightly lower emission intensity than that of the CN_m sample. This could be caused by the existence of defects in CN structure due to the incomplete CN polymerization using urea precursor [14, 15]. The other functional group such as C=O in the CN structure as confirmed in FTIR spectra of CN_u could be identified as one of such defects. It was reported previously that the defect played a role as an electron trap site in the photocatalyst structure [24]. In this case, when the photoexcited carriers were generated and be separated into electrons and holes, the electrons could be trapped in the surface defect and would not recombine with holes, resulting in the lower light emission. The lower emission spectra of the CN_u suggested a possibility to have better photocatalytic activity than CN_m since it represented a lower electron-holes recombination.

Photocatalytic Activity of CN

The photocatalytic activity of CN_u and CN_m was evaluated for the photocatalytic degradation of phenol. The percentages of phenol degradation are shown in Table 1. It could be observed that CN_u has 21.30% phenol degradation, which was 1.9 times higher than the phenol degradation of CN_m *i.e.*, 11.05%. This result showed that there was a favorable property in the CN_u samples to attain higher photocatalytic activity.

As discussed in the spectroscopy studies, the CN_m have lower bandgap energy than the CN_u. However, since the light source used was visible light with wavelength of more than 400 nm, such light source could be utilized efficiently by these two CN samples. Therefore, the bandgap energy was not the main reason for higher activity on the CN_u sample. On the other hand, the CN_u contained of C=O groups that could promote the electron localization and reduce the charge recombination. In this case, the presence of this defect seemed to be prominent as the reason for higher activity obtained on the CN_u.

TABLE 1. Properties and Phenol degradation on CN samples after 24 hours reaction under visible light irradiation

Samples	Phenol degradation (%)	Bandgap energy (eV)	Functional groups
CN _u	21.30	2.82	C-N, C=N, C=O, O-H, N-H
CN _m	11.05	2.62	C-N, C=N, O-H, N-H

CONCLUSION

Two precursors, namely urea and melamine, were used to prepare CN samples. The CN samples showed different physical colour appearances. The CN_u gave light yellow colour, while the CN_m sample gave intense yellow colour. The optical properties showed that the CN_u gave a lower absorption edge, and thus has a higher bandgap energy of 2.82 eV as compared to the CN_m with 2.62 eV. The FTIR and fluorescence spectra revealed that CN_u has C=O groups as the effect, which could act to reduce the electron recombination. The presence of this defect would contribute to the photocatalytic activity of the CN for phenol degradation under visible light irradiation. The CN_u exhibited 1.9 times higher photocatalytic activity than the CN_m sample. This study demonstrated that urea precursor was better than the melamine to obtain a highly active CN photocatalyst.

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