
Modification of Carbon Nanotube for Synthesis of Titania Nanotube (TiNT)-Carbon Nanotube (CNT) Composite

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Abstract

Carbon nanotube (CNT) is a material widely chosen for object of research in nano technology. Apart from its good absorbent property, CNT also has a unique structure, superior mechanic and electric properties and its high strength. The property of CNT above is to be expected to improve performance of Titania nanotube (TiNT) composite. Properties of pure CNT are hydrophobic at the surface and low dispersion stability. To improve dispersability of CNT then modification need to be modified. Adding Cetyl trimethyl ammonium bromide (CTAB) surfactant on CNT is a way to increase dispersion stability of TiNT-CNT. The objective of the research was to study influence of adding of Cetyl trimethyl ammonium bromide (CTAB) surfactant to synthesis of TiNT-CNT composite in degrading phenol compound. Pure CNT was added with CTAB surfactant in liquid, then was sonificated and dried. Surfactant Cetyl trimethyl ammonium bromide (CTAB) added CNT then composited with TiNT. Phenol degradation then tested by using TiNT-CNT (CTAB) in reactor for 4.5 hours. Then sample was characterized by employing Field Emission Scanning Electron Microscopy (FESEM), X-ray Diffraction (XRD), Fourier Transform Infra-Red (FTIR) and UV-vis Spectroscopy. The result of experiments from FESEM characterization showed forming of TiNT-CNT composites morphology. From X-ray Diffraction (XRD) characterization showed crystal formed on TiNT-CNT only of anatase crystal. Degradation of TiNT-CNT composite (CTAB) to phenol was also studied.

Keywords: Titania nanotube (TiNT)-Carbon nanotube (CNT) composite; Surfactant; Cetyl trimethyl ammonium bromide (CTAB); Phenol

1. INTRODUCTION

Photocatalysis process was occurred when photocatalyst and pollutant in contact. Most of photocatalyst semiconductor including TiO_2 has low adsorption, so that process of contact between photocatalyst and pollutant is not maximal and rate of recombination of electron is high with low utility [1], [2]. To overcome these drawbacks, photocatalysis by semiconductor photocatalyst need to be supported by an adsorbent as buffer [3]. In this case, TiO_2 samples is doped with non-metal elements, such as nitrogen, sulphur dan carbon to increase photocatalyst activity of visible light [4], [5]. Therefore, development to increase performance of photocatalyst TiO_2 is still becomes an interesting topic. By mixing photocatalyst and adsorbent, it is expected the contact between photocatalyst with pollutant become more effective. Further, used adsorbent is not require to be regenerated

because pollutant stucked on absorbent will degraded in situ by photo-catalyst so that saturation of absorbent can be avoided [6], [7].

Based on the previous research, it was known that the use of adsorbent as buffer can increase the rate of photo-degradation for various pollutants [6]. Used adsorbent was porous media, so that photocatalyst could get into pores or stucked on adsorbent surface.

Combination of photocatalysis-absorption is mixing of two processes with two materials which are photocatalyst (TiNT) and adsorbent (CNT) materials to produce composite material for various applications, one of them are pollutant material treatment. Producing of composite of adsorbent compound with photocatalyst make use some of additive chemical compound or modifier. Addition of the compound has an objective to increase the interaction between Titania and Carbon. Some interaction occurred regarding the addition, in some cases such as hydrophobic, interaction of $\pi - \pi$, interaction of electron-donor-acceptor (EDA), electrostatic interaction and hydrogen linking [8].

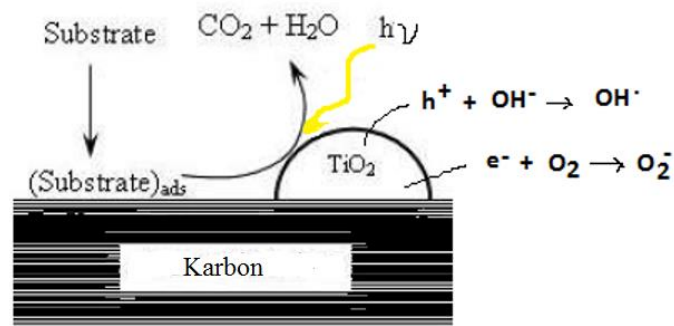


Figure 1.1 Illustration of combination of absorption-photocatalysis process

The composites consist of filler and matrix which produce bonding and acquired mechanical properties different from basic properties of each constituent component because interface between two constituent components. Interface is a surface built from bonding of matrix and filler for load transmitting. The interface could be inter atomic bonding, reaction inter matrix also can be strengthening of layer. The shape of composite morphology is based on surface modifications which are core cell, parallel and random.

The formation of composite can viewed from some approach, in which by functional group bonding and electron charge from the substance. The functional groups and electron charge could be formed thru treatment. One of the treatments is by acid and surfactant treatment. Surfactant surface could prevent grain formation during phase transformation and stabilize very small particle and make easy dispersion [9].

Adding of surfactant showed many bonding formed by other than covalent bonding. Surfactants acts as linking agent of surface adsorption of CNT by interaction of $\pi - \pi$. Surfactant has molecular structure consisted of hydrophilic and hydrophobic group. Hydrophilic group could be dispersed well in the water and organic solute, while hydrophobic group will make bonding with CNT.

Cetyl trimethyl ammonium bromide (CTAB) is one of surfactant from cationic type which could be used for modifying of CNT surface. Modified CNT by adding of CTAB is expected to increase dispersion of solution in synthesis of composites TiNT-CNT process, so that the TiNT-CNT composite could be formed.

The research was started with adding of CTAB in CNT. TiNT was synthesized from TiO₂ P25 and then continues to synthesis process of TiNT -CNT (CTAB) composites.

The general objective of the research was to acquire TiNT –CNT (CTAB) composite which has good performance of photocatalyst activity in degrading phenol.

In this research also developed a material engineering by combining adsorption – photocatalyst process on composites TiNT – CNT to acquire optimum parameter and condition in increasing of performance efficiency of composite material. Material engineering on this TiNT-CNT (CTAB) composite will be tested for effectivity of performance to eliminate phenol.

2. METHODOLOGY

CNT for the research was MWCNT type purchased from Dong Yang (HK) Int'l Group Limited of diameter 10-30 nm, length of 5-15 μ m, and purity of 99%, Cetyl trimethyl ammonium bromide (CTAB), TiO₂ (P25), NaOH, HCl, and aquades.

1.1 Synthesis of Titania nanotube (TiNT)

TiNT was synthesized by using hydrothermal process from TiO₂ (P25) added with NaOH 10 M in autoclave in temperature of 130 °C with stirring speed of 600 rpm for 6 hour, solution then rinsed with HCl and rinsed again by aquades until neutral pH. Then sample was dried.

1.2 Adding of surfactant Cetyl trimethyl ammonium bromide (CTAB) on Carbon nanotube

1 g of carbon nanotube (CNT) was added with CTAB surfactant of 0.5 g in 100 ml aquades. Solution was then sonicated for 1 hour. Then solution was dried.

1.3 Synthesis of Titania Nanotube (TiNT)-Carbon Nanotube (CNT) Composite

0.015 g CNT surfactant (CTAB) was added into 1 g of TiNT, the stirred by using magnetic stirrer for 3 hours. Solution then dried in temperature of 80°C, then mashed by pestle. Sample was then calcinated with in temperature of 400°C for 2 hour.

1.4 Performance test of TiNT-CNT composites using phenol compound

0.3 g composite sample TiNT-CNT was put into 300 ml of phenol liquid 10 ppm. The experiment was conducted for 270 minutes (4.5 hour), in which first 30 minutes was conducted without light (light off) and next 240 minutes the photodegradation using UV lamp as photon source (light on) was conducted. Photocatalyst used for the process was 0.3 grams powder and continuously stirred during experiment in order to make photocatalyst particles and phenol distributed evenly in sample solution. Quantitative result of degrade phenol was acquired by using spectrometer in accordance with procedure on SNI 06-6989.21-2004.

1.5 Characteristic

Sample was characterized by using FESEM, XRD, FT-IR and UV-vis spectroscopy.

3. RESULTS AND ANALYSIS

In Figure 3.1 (a-c) showed the morphology of CNT, TiNT and TiNT-CNT (CTAB) composite in pH 3. Figure 3.1 (a-b) also showed that morphology of CNT CTAB and TiNT was in tube shape. Figure 1c is morphology of TiNT-CNT (CTAB) composite and it showed that CNT CTAB was evenly distributed on TiNT matrix. Tube shape of TiNT and CNT can be seen formed random link. The link of tube formed composited so that adsorption-photocatalysis process able to perform synergy in conducting each task. This was supported by FT-IR result.

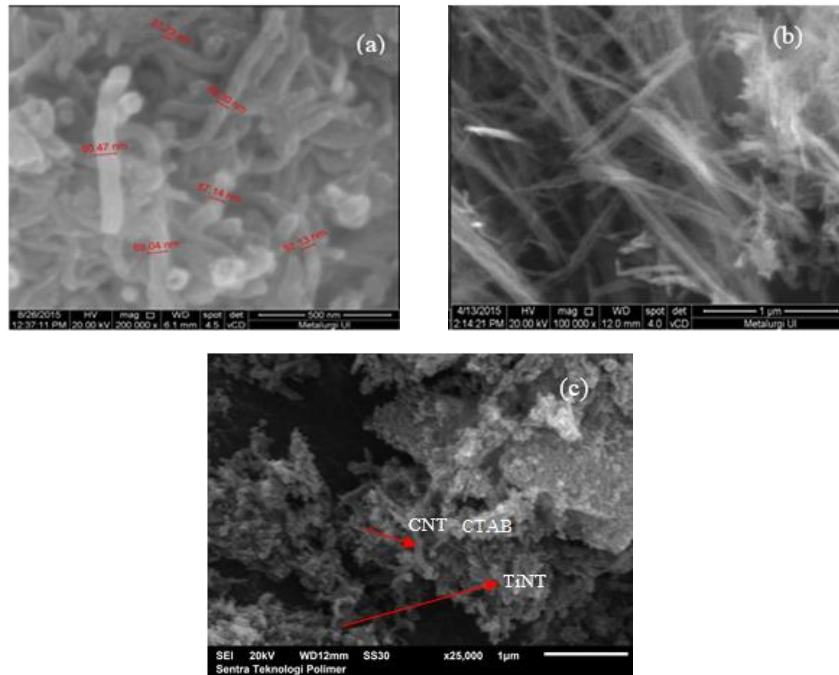


Figure 3.1 FESEM (a) CNT CTAB (b) TiNT (c) TiNT-CNT (CTAB) composite in pH 3

Figure 3.2 depicted photo-catalyst of TiNT-CNT composite have peak on of 2θ : 25.28°, 37.87°, 48.10°, 54.04°, and anatase 55.08°, This showed the existence of anatase crystal phase (JCPDS no. 21-1272). Photocatalytically, anatase structure showed better activity of reactivity compare with rutile structure [10]. Anatase structure is form frequently used because having bigger powder surface area and smaller particle compare with rutile structure and this structure occurred in range of heating temperature of decomposition of titanium compound (400°C-650°C).

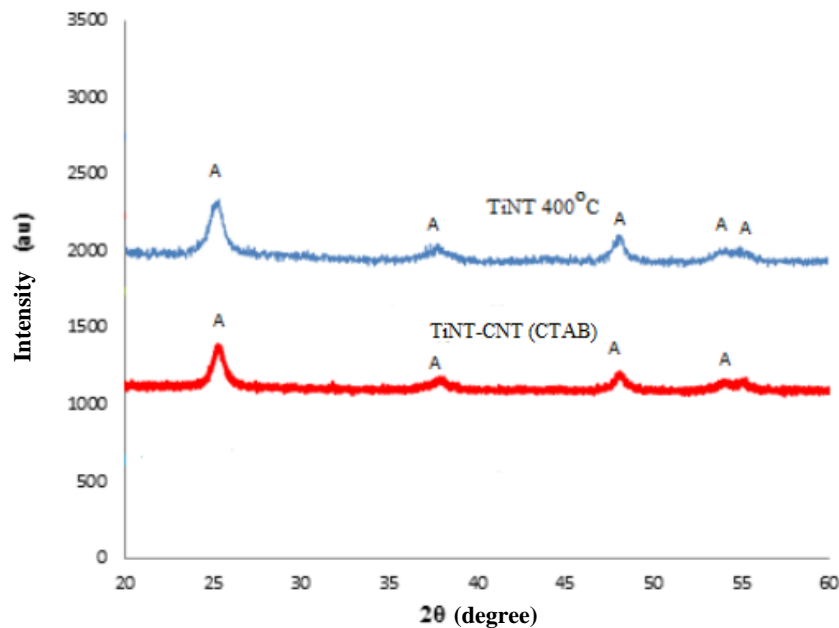


Figure 3.2 Analysis result of XRD TiNT and TiNT-CNT(CTAB) composite pH 3

Table 3.1 Size of Anatase Crystal

Catalyst	Size of anatase crystal (nm)
TiNT 400 °C	7.4
TiNT-CNT (CTAB)	9.8

Table 3.1 showed the size of anatase crystal (nm) of TiNT catalyst and TiNT-CNT (CTAB) composite. TiNT and TNT-CNT(CTAB) composite has crystal size of 7,4 nm and 9,8 nm consecutively. The shape of anatase crystal showed low level of catalyst reactivity under 10 nm. So that it much influenced the performance of catalyst in degrading phenol. It was showed in Figure 3.4 in which percentage of catalyst performance still low.

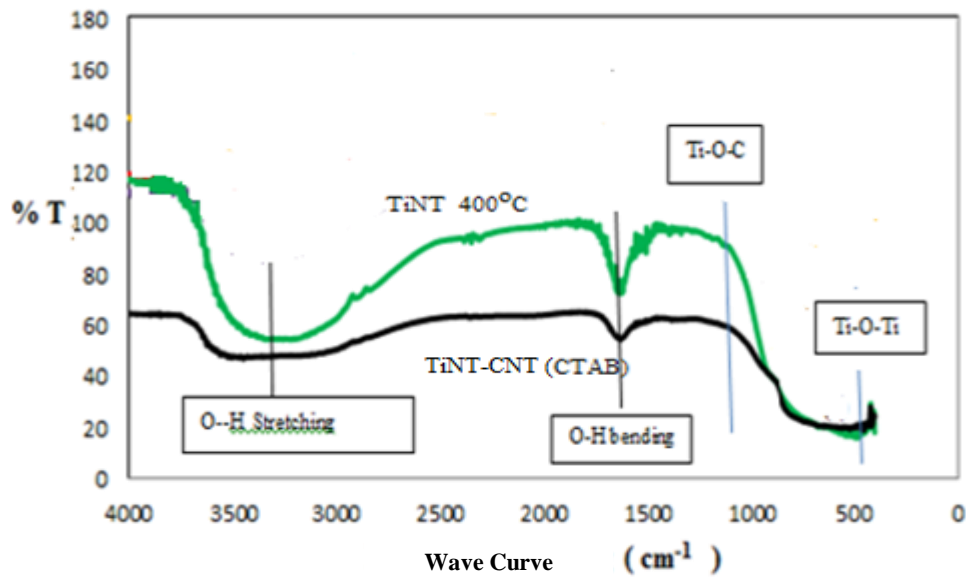


Figure 3.3 Results of FT-IR TiNT and TiNT-CNT (CTAB) composite

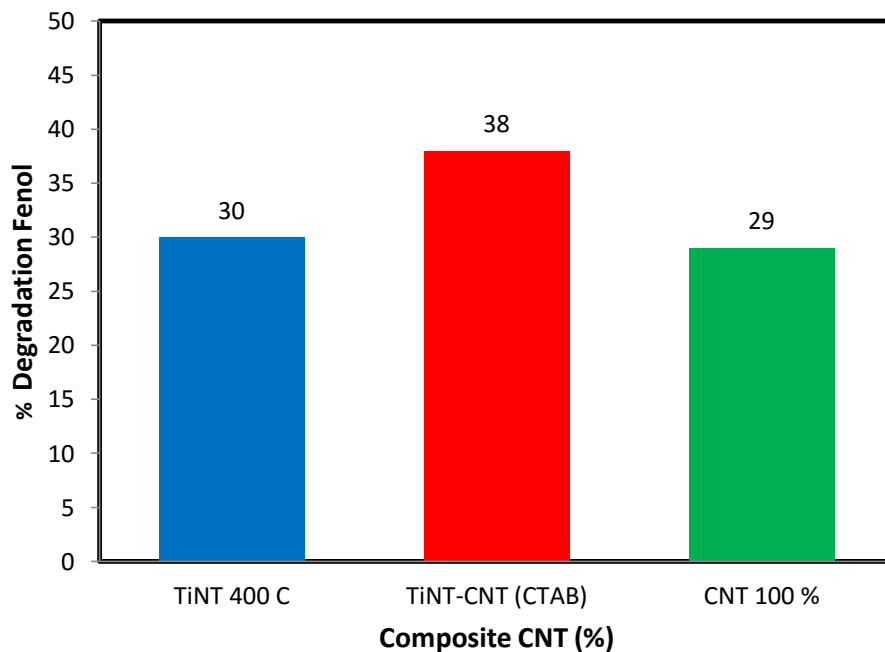


Figure 3.4 Effectivity of performance of TiNT-CNT(CTAB) composite to phenol degradation

The peak of available function groups in photocatalyst can be determined by FT-IR analysis as shown in Figure 3 below. Transmittance peaks showed in 420 and 840 cm^{-1} depict Ti-O-Ti stretching vibrations bond of TiO_2 crystal [11]. Carboxyl group which be marked by OH bond in carboxylate acid and spread to spectrum of 3400-2400 cm^{-1} identified as O-H stretching bond of OH bond to Ti atom [12]. Meanwhile, for all photocatalyst, the peak was around in 1560 cm^{-1} which constitute bending bond of surface containing hydroxyl group (surface which adsorb water molecules).

Figure 3.3 showed the performance of photocatalyst in degrading phenol from TiNT material, TiNT-CNT (CTAB) composite and CNT. Percentage of highest degrading phenol of performance of TiNT-CNT (CTAB) composite was 38 % higher than the one of individual performance of TiNT dan CNT CTAB. This showed that the objective of compositing of TiNT and CNT (CTAB) could be achieved in order to increase the effectivity of material performance in degrading phenol. But, it needs also considering other factors which have influence on degradation activity such as crystallinity, morphology and function group.

4. CONCLUSION

TiNT-CNT (CTAB) composite has been successfully synthesized by using Cetyl trimethyl ammonium bromide (CTAB) added CNT. Effectivity of performance of TiNT-CNT (CTAB) composite was influenced by morphology, function group and crystallinity. TiNT-CNT (CTAB) composite able to degrading phenol for 38 % (in 4,5 hour of experimental result).

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