A – INTRODUCTION

1. Rationale of the thesis

Today, the economy based on renewable hydrogen energy is becoming a new trend of development in the world and many countries are being called hydrogen economy. Hydrogen energy is a form of renewable energy in particular, and sensible use of all renewable energy sources in nature.

However, convert solar energy to hydrogen energy through the decomposition of water, in addition to solar energy and water, require photocatalyst based on nanostructured TiO_2. TiO_2 is selected as a photocatalyst with the following characteristics: suitable potential, photochemical activity durable and stable, including a portion of the light absorption spectrum of solar radiation spectrum, high photoconversion and high quantum efficiency, compatible with the the reaction medium, especially, low cost, ...

Many studies have been published, the usage of high-order TiO_2 nanotubes exhibit also great performance in water splitting under UV radiation (from 6.8 to 12.25%) due to nanotube structure properties. At the same time, modification of TiO_2 by "doping" transition metal ions, the nonmetal to extend visible light absorption edge. In which, methods non-metal anions and metal ions simultaneous doping exhibit remarkably effective. In particular, many studies show that, CdS doped TiO_2, the modified TiO_2 electrode exhibit high performance photoconversion by reducing the bandgap, reducing the recombination of photogeneration electrons and holes pairs while expanding the ability absorption of visible light in the photoelectrolysis.

2. Research objectives and contents

Fabricated nanostructured photocatalyst, ability absorption of visible light to apply for electrodes coatings in water photoelectrolysis into green hydrogen fuel.

The main research contents:

- Study on synthesis of TiO_2 nanotubes (TNTs) over chemical method (hydrothermal) and anodic oxidation method of Ti sheet (TNTA);
- Study on effects of dope agents on photoactivity of catalyst on TNTs powder to select appropriate agents;

- Study on fabrication of FTO photoanode covered catalyst based on TNTs by dipping and spin coating methods, and evaluate the effectiveness of these methods;
- Study on fabrication of electrode based on TiO_2 nanotube arrays (TNTA), the electrode is modified by the selected agents from above research;
- Evaluation of photocatalysis activity of photoelectrode in the water photoelectrolysis into hydrogen.

3. The scientific and practice meaning of the thesis

The thesis studied on synthesis and modification of catalysts based on TiO_2 nanotubes via different methods, which highlights, the CdS/TNTA-20 catalyt is fabricated (TNTA be synthesized by electrochemical method and CdS modified after 20 immersion cycles). The catalyst exhibit high activity in water photoelectrolysis reaction into hydrogen.

4. The new contributions of the thesis

- Examined systematically and successfully synthesized TNTA by the method of anodized Ti foil in glycerol electrolyte solution containing NH_4F at 40V for 8 hours. The results showed that obtained TiO_2 nanotubes with length of 3 μm, diameter of 130 nm;
- Successfully synthesized Pt/rGO/TNTs nanocomposite catalysts by hydrothermal method. The charaterization results clearly show the role of Pt and RGO in reducing the recombination of photogeneration electron/ holes pairs, enhance photocatalytic performance of TNTs;
- Photoactivity evaluated results of catalyst coated electrode in MB photodecomposition reaction showed catalyst Pt/rGO/TNTs coated electrode exhibie higher photochemical activity. This has opened up application trends of Pt/rGO/TNTs catalyst coated electrode in water photoelectrolysis reaction into hydrogen.
- Systematically studied on fabrication method CdS doped TNTA electrode and identified: 20 immersion cycles, CdS/TNTA-20 electrode exhibit the best photochemical performence.
- Surveyed and evaluated the stability of the CdS/TNTA-20 electrode in the water photoelectrolysis reaction. The result shows that, in the

S^{2-} ion containing electrolyte solution, operations of this electrode are more stable and durable than these in the conventional KOH electrolyte solution. The photocatalytic activity in the photoelectrolysis shows that, the amount of hydrogen generated reached 1.5 ml/h/cm^2, is 5.6 times higher than this of TNTA electrode.

5. Layout of the thesis

The thesis consists of 98 pages (excluding references) divided into sections: Introduction of 3 pages, Chapter 1 Overview of 30 pages, Chapter 2 Experiment of 14 pages, Chapter 3 Results and Discussion of 49 pages, Conclution of 2 pages. The thesis consists of 6 tables, 66 pictures and photos and charts, 138 references.

B – MAIN CONTENT

CHAPTER 1: OVERVIEW

This chapter presents an overview of the water photoelectrolysis process, photocatalysis based on TiO_2 nanotubes (TNTs). TNTs synthetic methods, the modified methods, the fabrication methods of electrode apply for organic compounds photodecomposition and water photoelectrolysis into hydrogen.

CHAPTER 2: EXPERIMENT

The experiments are performed in National Key Laboratory for Petrochemical and Refinery Technologies – Viet Nam Institue of Industrial Chemistry and University of Science and Technology – The University of Danang.

2.1. Preparation of catalysts

- **Preparation of TNTs**: TNTs was synthesized by hydrothermal method, TNTA was synthesized by method of anodized Ti foil in glycerol electrolyte solution containing NH_4F.
- **Metals and metal oxides doped TNTs**: Modified TNTs by wet impregnation with 1% weight metals and metal oxides (Fe, Cu, …).
- **Non-metalic doped TNTs:** C doped TNTs from the GO precursor, N doped from with urea by hydrothermal alkali, the product was washed, filtered, dried, annealed.

- **Mixture doped TNTs:** CdS TNTs by wet impregnation, Pt/rGO doped TNTs by hydrothermal method, the product was washed, filtered, dried, annealed.
- **CdS doped TNTA:** CdS doped TNTA by the successive ionic layer adsorption and reaction (SILAR) process, the product was washed, filtered, dried, annealed.

2.2. Fabrication of electrde

Fabrication of adhesives based on TiO_2 sol. Fabrication of TNTs powder coated FTO electrode fabricated by dipping and spin coating methods. Fabrication of TNTA electrode by method of anodized Ti foil.

2.3. Test catalytic activity

The photoactivity was evaluated in the methylene blue (MB) photodegradation reaction and the water photoelectrolysis in National Key Laboratory for Petrochemical and Refinery Technologies.

2.5. The catalytic characteristic methods

The catalysts were characterized by the modern physical and chemical methods such as: X-ray diffraction (XRD), Transmission electron microscope (TEM), Scanning electron microscope (SEM), Scanning electron microscopy combine with Energy-dispersive X-ray spectroscopy (SEM-EDX), Field emission scanning electron microscopy (FE-SEM), ultraviolet-Visible spectra (UV-Vis), Photoluminescence spectra (PL).

CHAPTER 3: RESULTS AND DISCUSSION

3.1. Synthesis and characteristic properties of TiO_2 nanotubes (TNTs)

3.1.1. Synthesis by hydrothermal method

The phase properties characterized results by X-ray diffraction (XRD), and the microstructure properties by transmission electron microscope (TEM) of TNTs showed in fig.3.1 and fig.3.2.

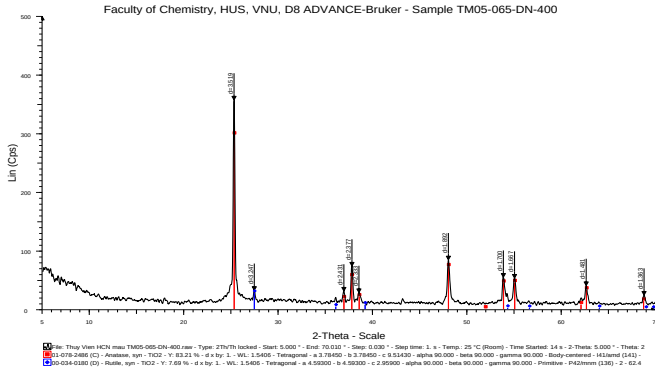
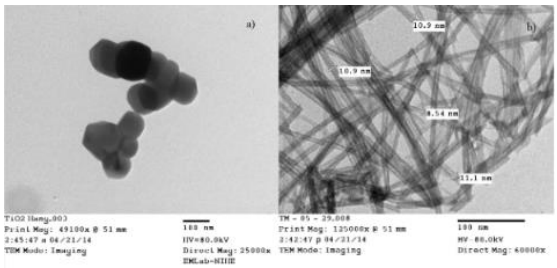


Fig. 3.1: XRD schema of TNTs, annealed at 400°C



Hình 3.2: Ảnh TEM của mẫu TiO_2 TM (a) và TNTs (b)

Fig.3.1 shows that, after thermal treatment at 400°C, on the TNTs XRD schema is only the typical peak of anatase phase.

The results in Fig.3.2 shows, commercial TiO_2 powder raw materials has completely transformed into TiO_2 nanotubes with diameter ranges of 8-11 nm.

3.1.2. Synthesis by anodized

3.1.2.1. Effect of current density

The survey result of current density change according to voltage and time in fig.3.3 shows that, in each of the different voltage values, current density was changed strongly during first time and reached stable after about 3 hours. Therefore, voltage was selected to survey effects of voltage on the morphology and structural of tubes.

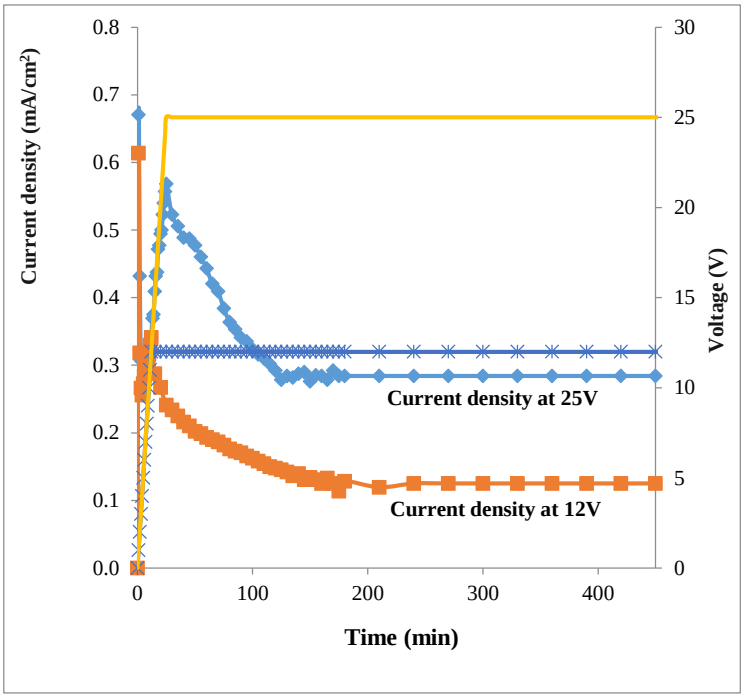


Fig. 3.3: Current density at different times and voltages

3.1.2.2. Effect of anodized time

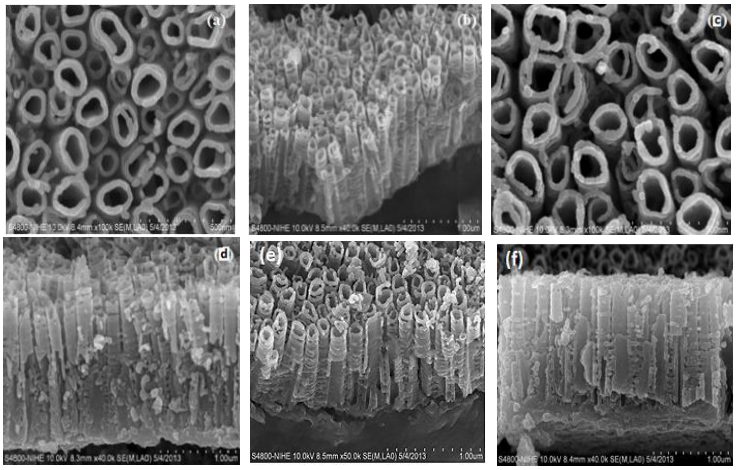


Fig. 3.4: SEM image of TNTs-5-25 (a, b); TNTA-8-25 (c, d); TNTA-12-25 (e); TNTA-16-25 (f)

The obtained results in Fig.3.4 show that, formed nanotubes TiO_2 is vertical and relatively uniform. Length increase from 0,8 µm to 1,18 µm and diameters increase from 70 nm to 100 nm duration time 5 to 8 hours, respectively (Table 3.1). As anodized time continuously increase (12, 16 hours), as well as length and diameter increase slightly. Thus, 8 hours is the appropriate anodized time and this value was applied for subsequent studies.

Table 3.1: Size of TiO_2 nanotubes at different anodized times

No	Samples	Length (µm)	Diameter (nm)	Thichness (nm)
1	TNT-5-25-550	0.80	70	42
2	TNT-8-25-550	1.18	100	42
3	TNT-12-25-550	1.26	113	44
4	TNT-16-25-550	1.38	123	47

3.1.2.3. Effect of potential

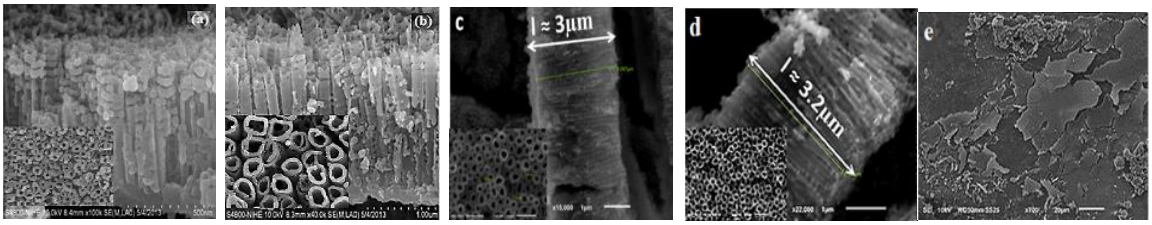


Fig. 3.5: SEM image of TNTA at 12V (a), 25V (b), 40V (c), 50V (d), 60 (e)

The research results shows that, potential increases from 12V to 25V, the diameter increases from 50 nm to 130 nm, the length increases from 440 nm to 1.180 µm. The voltage increases to 50V, the length and diameter increase slightly (3,2 µm and 140 nm). However, potential up to 60V, tube structure disappears. Thus, anodization applied potential of 40V is appropriate and this value applied for subsequent studies.

3.1.2.4. Effect of solvent

At an applied potential of 40V, nanotubes array formed in ethylene glycol solvent (EG5), which is 3 times longer than the one in glycerol solvent (G5). However, obtained diameter of tube in glycerol solvent is larger than the one in EG.

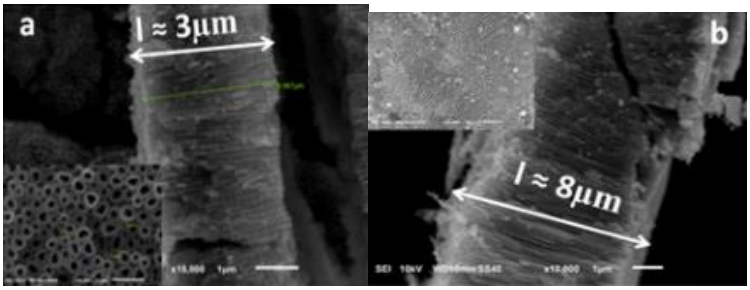


Fig. 3.7: SEM image of TNTA in G5 (a); EG5 (b)

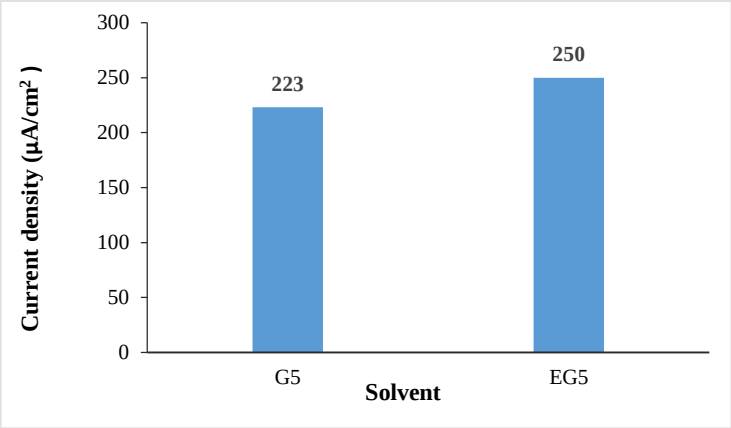


Fig. 3.8: Current density of TNTA samples at diffrent solvents

The photoelectrochemical test results by determining photocurrent of samples show that, the photoelectrochemical performance of TNT-8-40-EG5 and TNT-8-40-G5 is similar (250 µA/cm^2 and 223 µA/cm^2). Thus, glycerol is suitable solvent for the anodization.

3.1.2.5.Effect of thermal treating

The result in Fig.3.9 shows that, at below calcination thermal, on the XRD schema is only the typical peak of anatase phase. Temperature increase (500ºC, 550ºC, 600ºC và 650ºC), the observed result appear the typical peak of rutile phase.

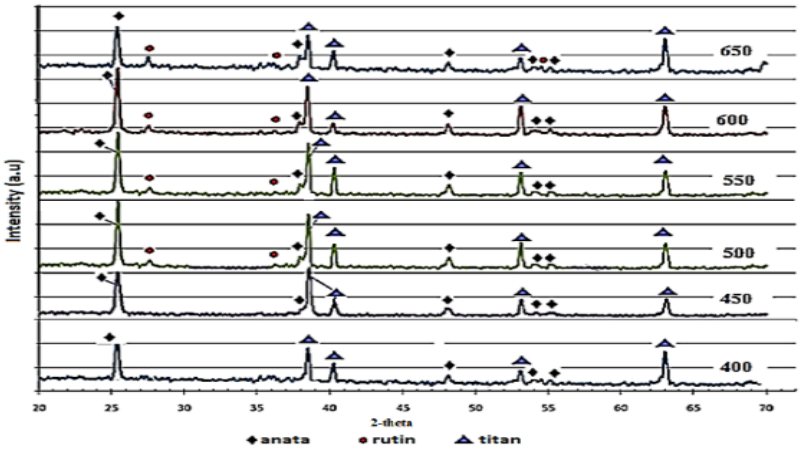


Fig. 3.9: XRD schema of samples at different thermal treating

Anatase/Rutile ratio decreased with increasing temperature from 550-600°C, which is attributed to the transformation a part of Anatase phase to Rutile when the temperature increases. Thus, 450°C is the is appropriate calcination temperature.

3.2. Study on doped TiO_2 nanotubes and survey the photocatalytics activity

Hydrothermal synthesized TNTs powder was selected as the object to study on effect of different dope agents on photocatalysis activity.

3.2.1. Metal doped TNTs

Figure 3.11 present the evaluation results of the catalytic activity in MB photodecomposition reaction.

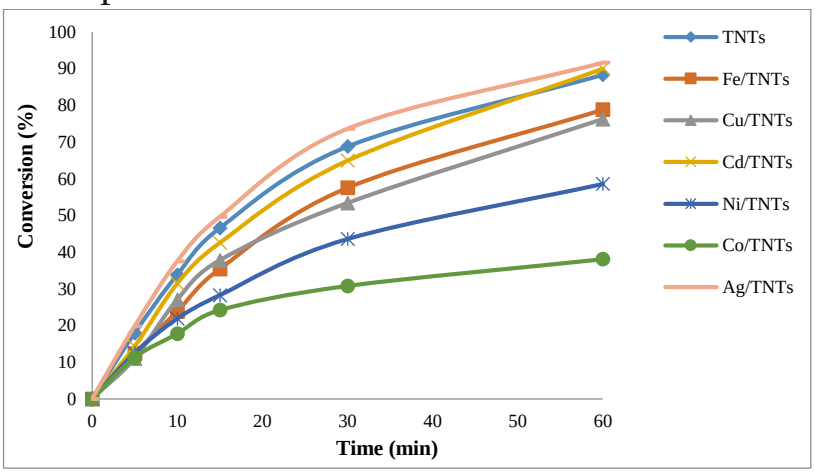


Fig. 3.11: Conversion of MB over Metal doped TNTs

The result in Fig.3.11 shows that, after 60 min, metal doped TNTs exhibit high MB conversion, 88%. The conversion of modified samples ascending order as follows: Co/TNTs, Ni/TNTs, Cu/TNTs, Fe/TNTs, Cd/TNTs, Ag/TNTs, corresponding to the value of 38%, 58%, 76%, 78,8%, 90%, 91,6%. Compared to TNTs, the conversion of metal doped samples is not much improved.

3.2.2. Metal oxide doped TNTs

The result in Fig.3.13 shows that, dopping with the metal oxides did not improved the photocatalytic performence of TNTs in MB photodecomposition reaction.

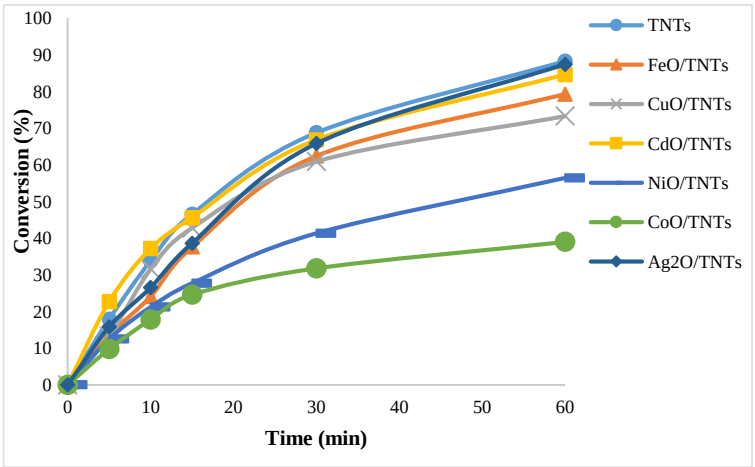


Fig. 3.13: Conversion of MB over Metal oxides doped TNTs

3.2.3. Non-metalic doped TNTs

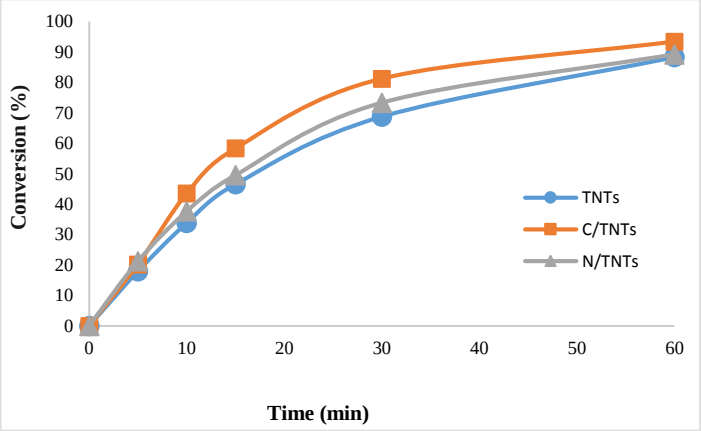


Fig. 3.15: Conversion of MB over Non-metalic doped TNTs

The result in Fig.3.15 shows that, the conversion of N, C doped samples exhibit high photocatalytic in MB photodecomposition reaction. But, the diffrence between them is not much. Thus, the non-metallic dopping is not much improved photoactivity of TNTs.

3.2.4. Mixture doped TNTs

The result in Fig.3.17 shows that, the MB conversion of Pt/rGO/TNTs, CdS/TNTs samples are similar, corresponding to the value of 93,8% and 95,4%, higher than the one of original TNTs.

From the above research result shows that, TNTs powder doped with CdS and Pt/rGO display the best photochemical efficiency. Thus, the Pt/rGO, CdS catalysts was selected to investigate and charaterize in detailly.

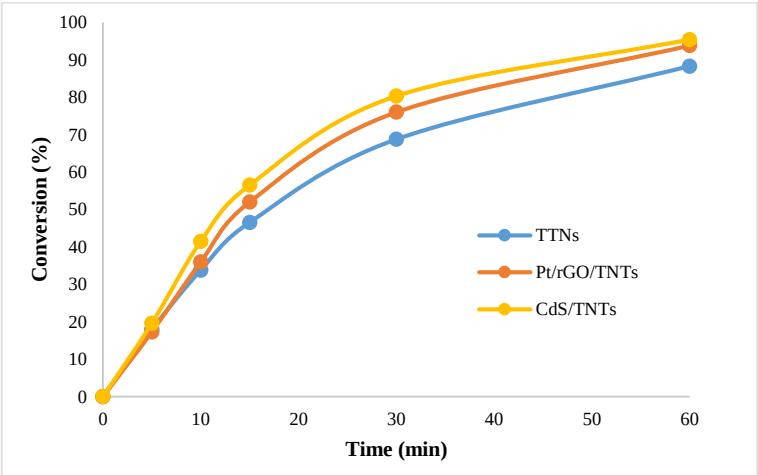


Fig. 3.17: Conversion of MB over Mixture doped TNTs

3.3. Study on synthesis and charaterization of Pt/rGO/TNTs composite

The phase content analysis results of the Pt/rGO/TNTs samples in Fig 3.18 show that, after dopping, the sample has the typical peak of anatase phase, and in the presence of Pt and no detected characteristic of the GO at $2\theta = 10^{o}$. This proves that, the GO completely reduced to rGO.

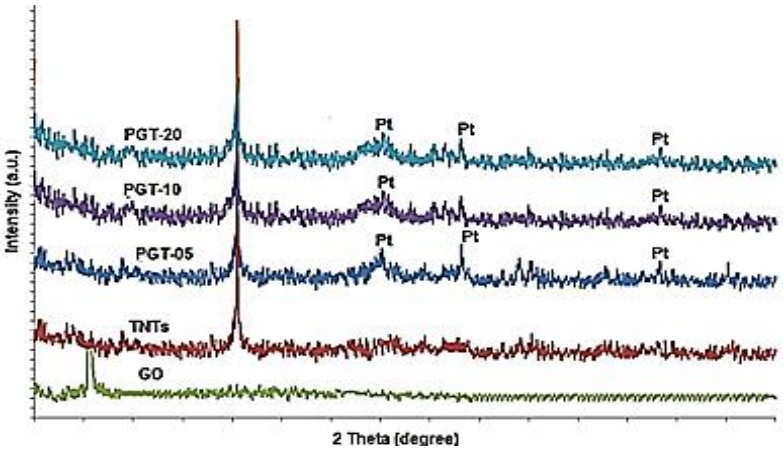


Fig. 3.18: XRD schema of different rGO amounts

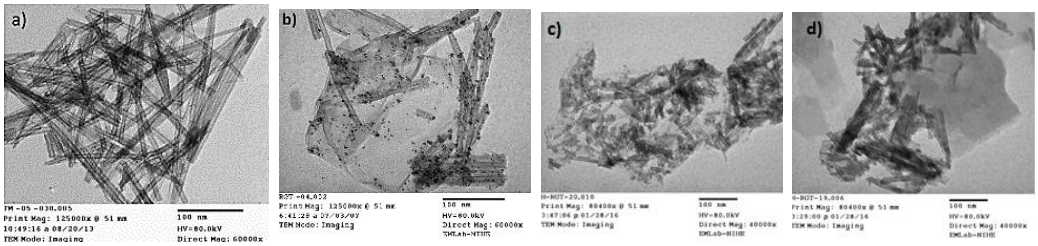


Fig. 3.19: TEM images of samples: a) TNTs, b) PGT-10, c) PGT-05, d) PGT-20

The TEM images in Fig. 3.19 b show that, the nanoparticles Pt and TNTs were well dispered onto GR sheets. A lower weight ratio of GO (5%), there was no appreciable present of GR sheet (Fig. 3.19 c). A higher weight ratio of GO (20%) to TNTs in PGT-20, GO was not well dispersed and Fig. 3.19d shows that the rGO sheet was made the clusters and not uniformly covered TNTs and Pt NPs. Thus, GO at 10% is appropriate.

The result in Fig.3.20 shows that, the appearance of the elements Pt, C, O, Ti with C and Pt content of 48,5% and 0,95%, respectively.

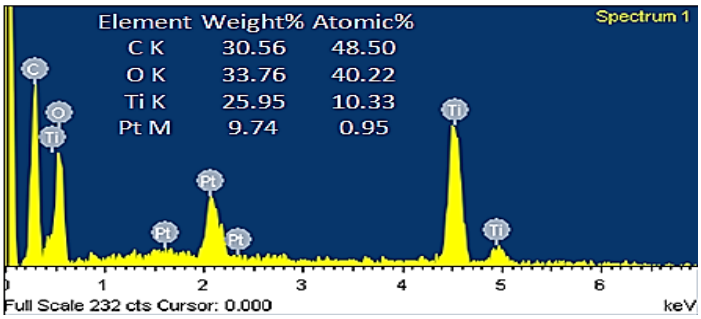


Fig. 3.20: EDS spectra of PGT-10

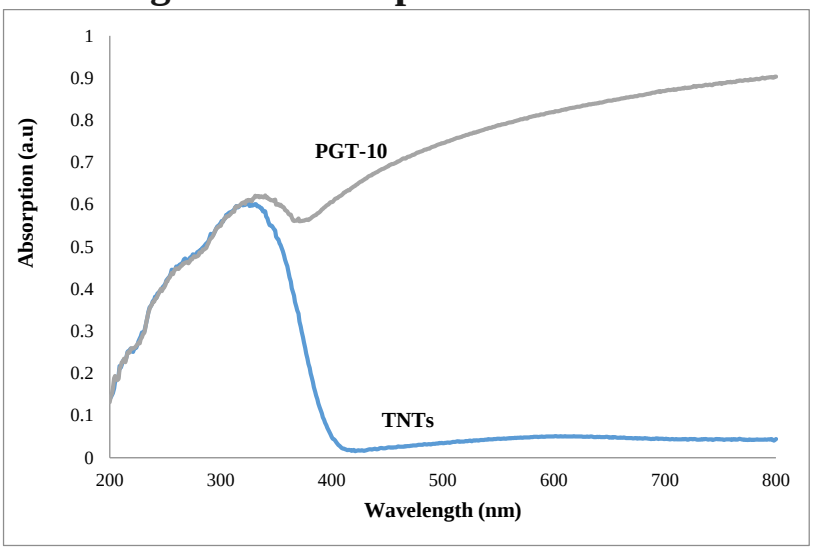


Fig. 3.21: UV-Vis spectra of TNTs and PGT-10

The result in Fig.3.21 shows that, compare to original TNTs, the absorption band edge of PGT-10 was red-shift and extended to the visible region.

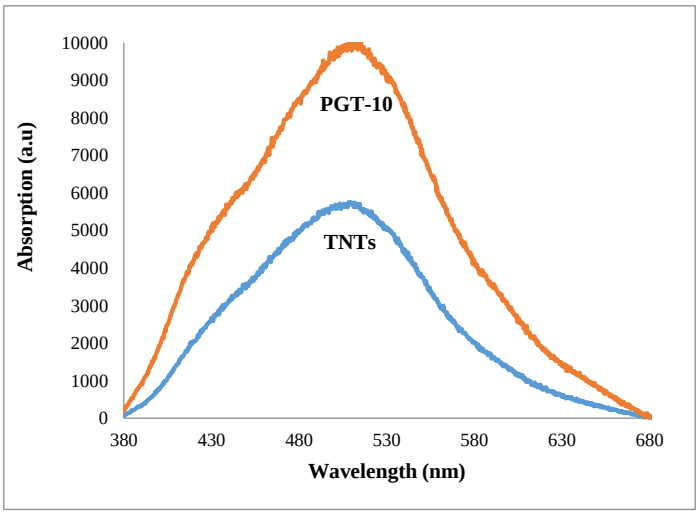


Fig. 3.22: PL spectra of of TNTs and PGT-10

The result in Fig.3.22 shows that, PGT-10 exhibit lower emission intensity, suggested that, the simultaneously dopping Pt and rGO enhanced the absorption band edge to visible light region.

From above research results by morden characteristic technicals demonstrated that, the role of Pt and rGO in Pt/rGO/TNTs catalyst nanocomposite, photocatalytics performance of original TNTs was improves. This result suggested that a great potential for applications in the water photoelectrolysis.

3.4. Study on catalyst coating method

3.4.1. Study on catalyst coating by dipping method

3.3.1.1. Study on effect of sol composition

Table 3.3: Effect of sol composition

No	Mixture sol	Dipping rate (cm/min)	Comment
1	1	4	Electrode surface is cracked, the film is peeled.
2	2 and 2-2		Electrode surface is cracked, the film is peeled.
4	3		Electrode surface is smooth, transparent films, good adhesion, no peeled.
5	4-1 and 4-2		Electrode surface is cracked, the film is peeled.

The result in Table 3.3 shows that, in the similar dipping rate, in the sol-gel solutions using ethanol with difference ratio, the 3rd sol is suitable composition to creat transparent film and good adhesion on the FTO electrode surface and thermal stability. Thus, the 3rd sol solution was applied for subsequent studies.

3.4.1.2. Study on effect of dipping rate

The survey results effect of dipping rate on the formation of catalyst film on the FTO electrod using 3rd sol shows that, 4 cm/min is suitable dipping rate and this value applied for subsequent studies.

Table. 3.4: The survey results effect of dipping rate

No	Dipping rate (cm/min)	Comment
1	1	Electrode surface smooth, the film is slightly thin
2	2	
3	4	Electrode surface smooth, the film exhibit good adhesion.
4	6	Electrode surface is relativity smooth, slightly stacked, the film is peeled after immersion in water
5	8	Electrode surface is stacked, the film is peeled after thermal treatment.

3.4.1.3. Study on effect of number of dipping times

The result in Fig. 3.26 shows that, the number of dipping time increases, the weight of TiO_2 on electrode significantly increases. However, the weight of TiO_2 film increases according to the number of dipping times, respectively, thickness of film increases cause stacking, pitting (Fig. 3.27). The cracks, cracked surface increases proportional to the number of dipping times.

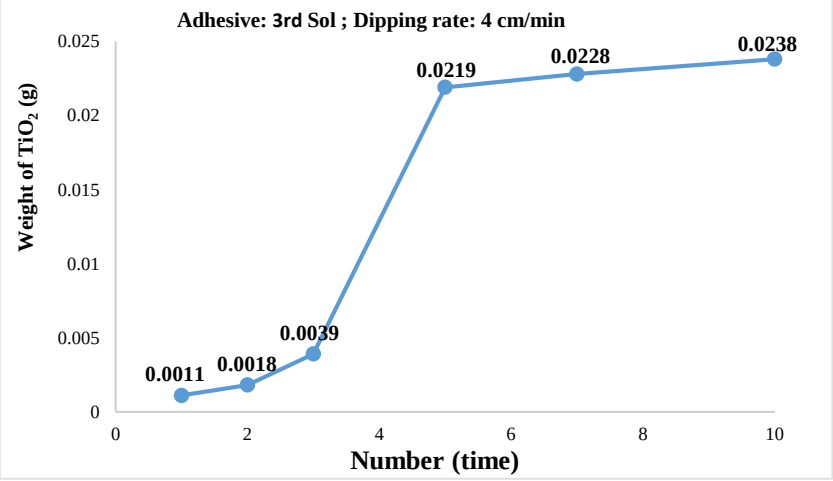


Fig. 3.26: Effect of the number of dipping times

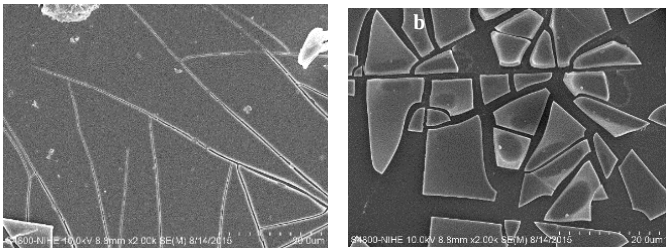


Fig. 3.27: SEM image of 3 (a) and 5 dipping times (b)

Although, dipping method made a transparent, thin film on the electrode surface with good adhesion. However, the limitation of dipping method is only taken a limited amount of catalyst on electrode surface due to could not repeated many times because the surface will be cracked, broken when coating thickness increase.

3.4.2. Study on spin coating method

3.4.2.1. Study on effect of sol composition

The preliminary assessment results of effect of the sol composition to adhesion on the electrode surface by spin coating method [95, 97] are presented in Table 3.5.

The result shows that, among the samples using ethanol is only the 3rd sol has matching composition, creating transparent film, good adhesion on the electrode FTO surface.

Table 3.5: The survey result effect of sol composition

No	Sol mixture	Comment
1	1	The film is peeled
2	2 and 2-2	The film is peeled
4	3	Electrode surface smooth, the film exhibit good adhesion.
5	4-1 and 4-2	Electrode surface is stacked, the film is peeled

3.3.2.2. Study on effect of thermal treating

The analytic result of the Pt/rGO/TNTs catalyst coated electrode at different treating temperature in the XRD schema shows that, at 350^{o}C, 450^{o}C, is only characteristic peak of anatase phase. Temperature

increases to 550°C, the appearance of phase transformation from anatase to rutile. Thus, the temperature at 450°C is appropriately treatment.

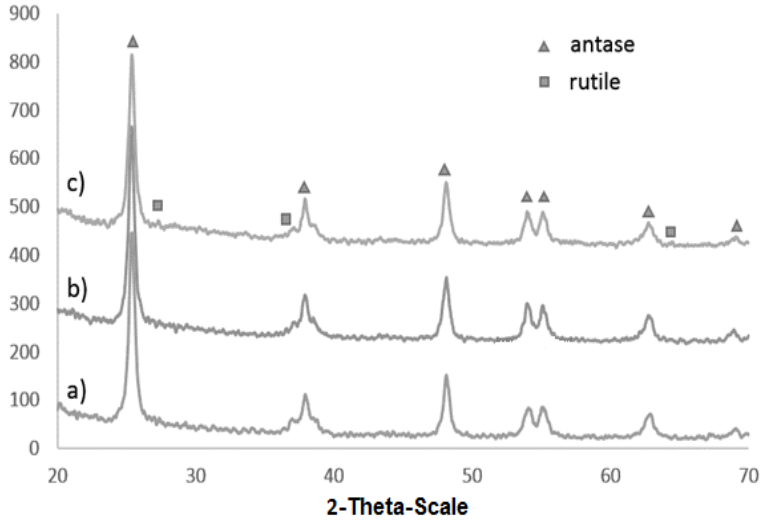


Fig. 3.30: XRD schema of 350°C, b) 450°C and c) 550°C

3.4.3. Study on stability between catalyst film and electrode surface

3.4.3.1. Assessment of bonding strength

The result in Table. 3.6 shows, after one day, the electrde was immersed in electrolyse solution, amount of titanium was dissolved of 0.002 mg is accounted for 0.1667% coating film weight (coating weight of 1.2 mg). After 10 days, amount of titanium was dissolved of 0.242%. This result shows that, although the film has good adhesion to the electrode surface, but a litle amount of TiO_2 was dissolved into the electrolyse environment.

Table. 3.6: Adhesive strength of film

No	Time (days)	Ti dissolving (mg)
1	Before	0
2	1	0,0020
3	2	0,0023
4	3	0,0024
5	6	0,0026
6	10	0,0029

3.4.3.2. Assessment of photocatalytic stability

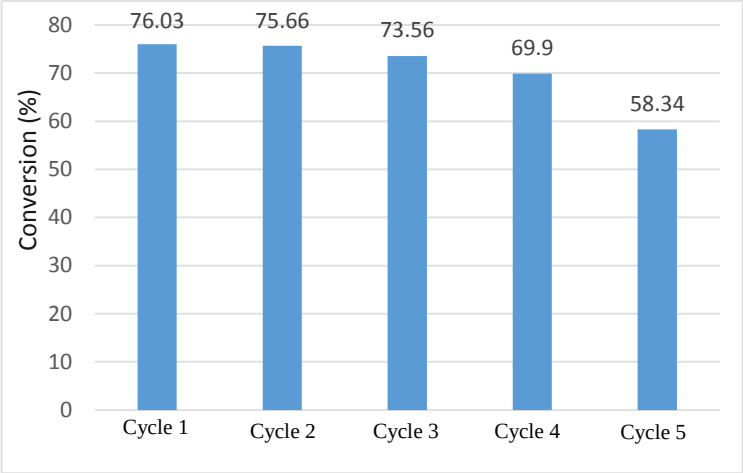


Fig. 3.33: Photocatalytic stability of Pt/rGO/TNTs coated electrode

The result shows, MB conversion slightly decrease after two cycles. However, the next cycles, MB conversion clearly decrease and down to 58,34% in cycle 5.

Thus, Pt/rGO/TNTs catalyst coated electrode exhibit high MB conversion. However, stability of the electrode in the reaction environment should be improved.

3.5. Study on characterization and photocatalytic test of modified electrode on Ti metal substrate

3.5.1. Strength of TNTA electrode

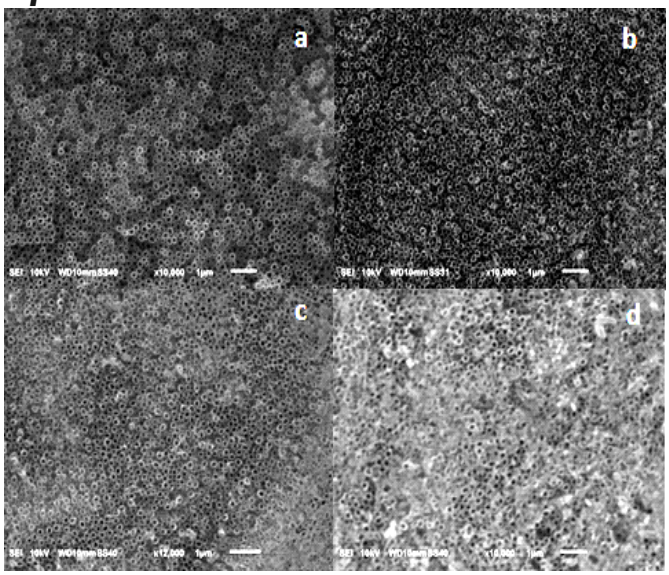


Fig. 3.34: SEM image of TNTA during the immersion time a) 1 day; b) 3 days; c) 5 days; d) 10 days;

The microstructure characteristic result shows that, the electrode surface was unchanged after 10 days immersed in electrolyse solution, which demonstrated the coating exhibit high adhesion to the Ti metal electrode.

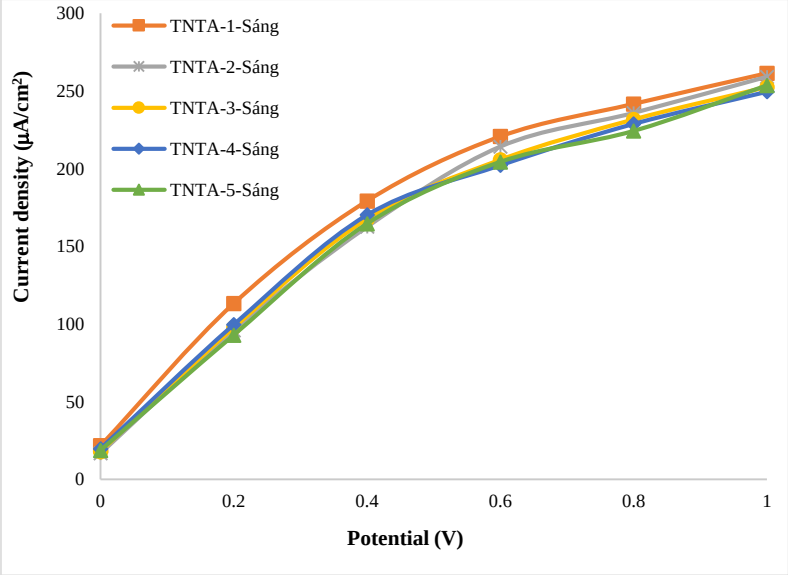


Fig. 3.35: Active stability of electrode in photoelectroysis reaction

The result shows, at an applied potential of 1V, the current density of TNTA electrode was no significant differences aftet 5 cycles (261,3 µA/cm^2 in cycle 1 and 253,5 µA/cm^2 in cycle 5).

3.5.2. Effect of treatment process

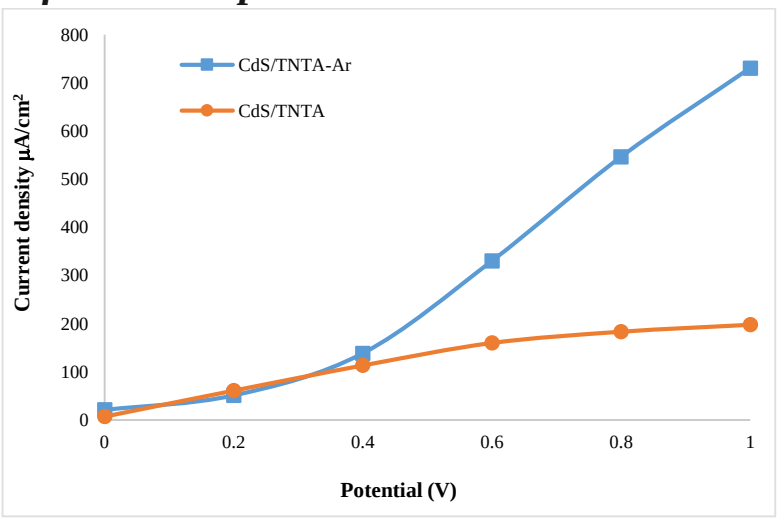


Fig. 3.36: Current density of catalyst before and after calcination

The result in Fig.3.36 shows that, at applied potential of 1V, the samples clearly present photocatalytic activity, the obtained current density was significant high. The current density of argon annealed

sample was 730,2 µA/cm^2 is 3,7 times higher than the one obtained by non annealed sample (197,2 µA/cm^2).

3.5.2. Effect of immersion cycle

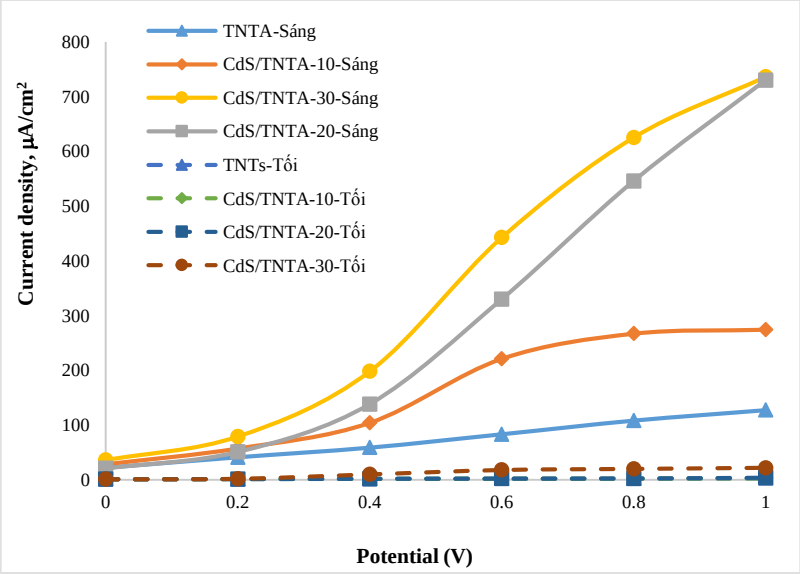


Fig. 3.37: Current density of samples at different cycles

The measured photocurrent density result in Fig. 3.32 shows, the samples exhibit good photocatalytic activity, in illumination condition. As potential increases, the photocurrent density increases. However, after 30 CdS deposition immersion cycles, the photocurrent density increases not much (at 1V). Evaluate the ratio of photocurrent in illumination and dark condition, the CdS/TNTA-30 sample is the least value. Thus, the photocurrent density of the CdS-doped TNTA sample is the highest after 20 deposition immersion cycles, and this catalyst was selected to use as electrode in water photoelectrolysis.

3.5.4. Characterization properties of CdS/TNTA-20 electrode

The SEM image in Fig 3.40 shows that, the CdS doped sample exhibit precipitate with different morphology on the surface, on the mouth of the tubes and the inside tubes. After annealing, nano-sized CdS particles was formed.

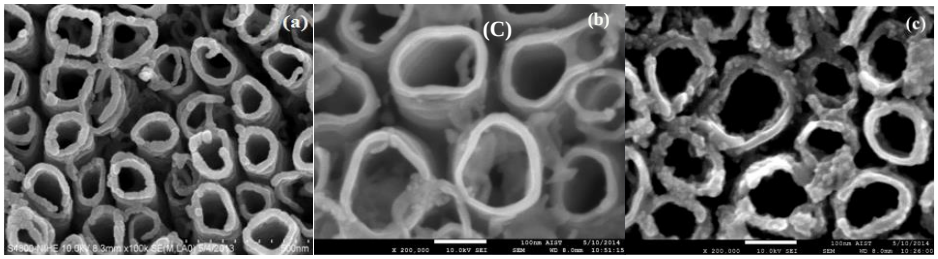


Fig. 3.40: FE-SEM image of (a) TNTA; (b) CdS/TNTA-20 annealed (c)

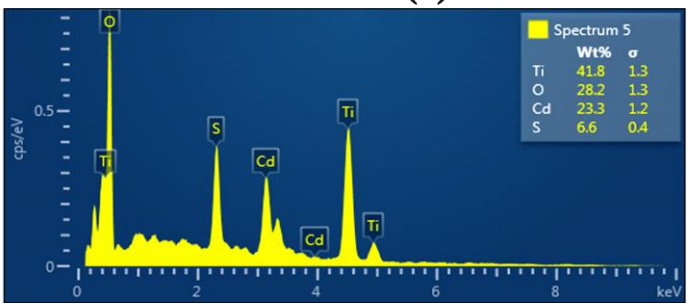


Fig. 3.41: EDX spectra of CdS/TNTA-20 electrode

The composition analytic result of titanium, oxygen, cadmium and sulfur on the CdS/TNTA-20 sample by EDX method shows that: 41,8%; 28,2%; 23,3% and 6,6%, respecticely, with a molar ratio of Ti/O ~ 1/2 and Cd/S ~ 1/1. This result demonstrated that, the method used in the preparation of CdS-doped TNTA in this thesis is suitable.

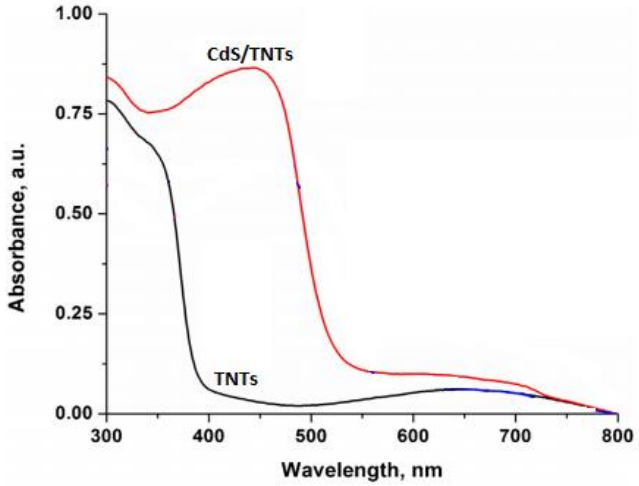


Fig. 3.42: UV-Vis spectra of CdS/TNTA-20 and TNTA samples

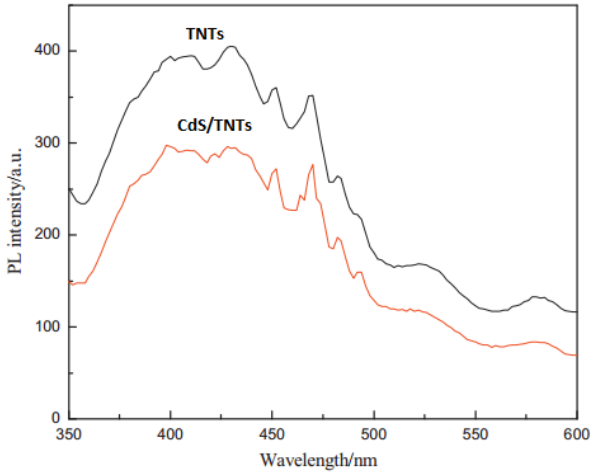


Fig. 3.43: PL spectra of CdS/TNTA-20 and TNTA samples

The result in Fig. 3.42 show that, the absorption band edge of CdS/TNTA-20 was extended to the visible light region with absorption edge arrange 540 nm and absorption peak arrange 420 nm.

The result in Fig. 3.43 shows that, compare to original TNTA, emission intensity of CdS/TNTA-20 decreases. This may explain that, the band gaps and recombination of photogeneration electrons and holes of TNTA decrease.

3.5.5. Photocatalytic activity test of CdS/TNTA-20

The result in Fig. 3.45 shows that, at 1V, the photocurrent of CdS/TNTA-20 electrode is much higher than this of TNTA. This result also proves, CdS improved photocatalytics performance of TNTA electrode under illumination condition.

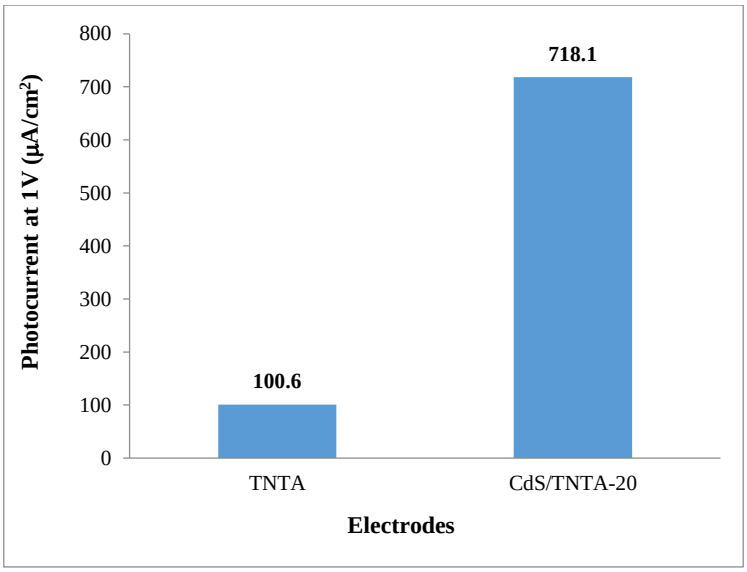


Fig 3.45: Photocatalytic activity of electrodes

3.5.6. Stability test of CdS/TNTA-20 electrode

The result in Fig. 3.46 shows that, at 1V, the photocurrent of CdS/TNTA-20 electrode, in KOH electrolyte solution, decreases duration time and equivalent to the value of TNTA. The photocurrent of CdS/TNTA-20 electrode is high stability in $(NH_4)_2S$ solution. This is similar to the results published by the Yanbiao Liu et al [46].

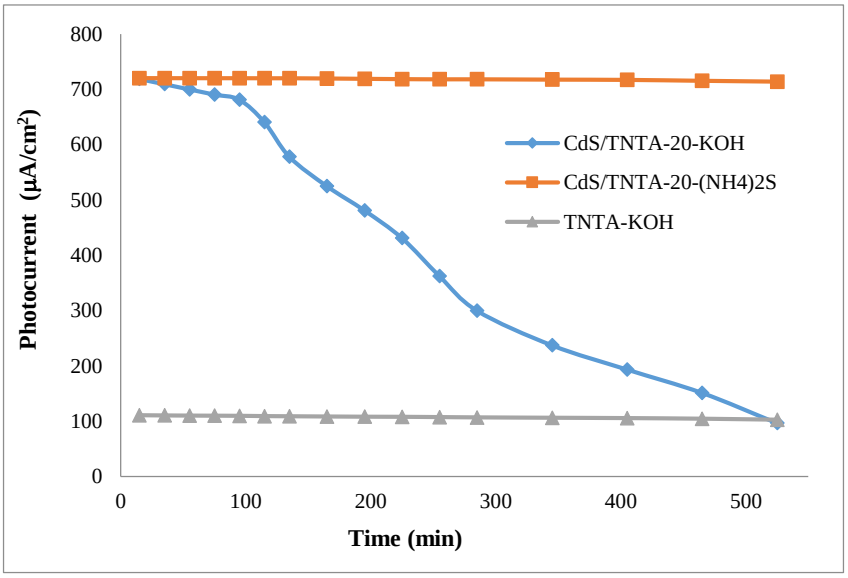


Fig. 3.46: Lifetime of CdS/TNTA-20 in different electrolyte solution

3.5.7. Photocatalytic activity of CdS/TNTA-20 electrode in water photoelectrolysis

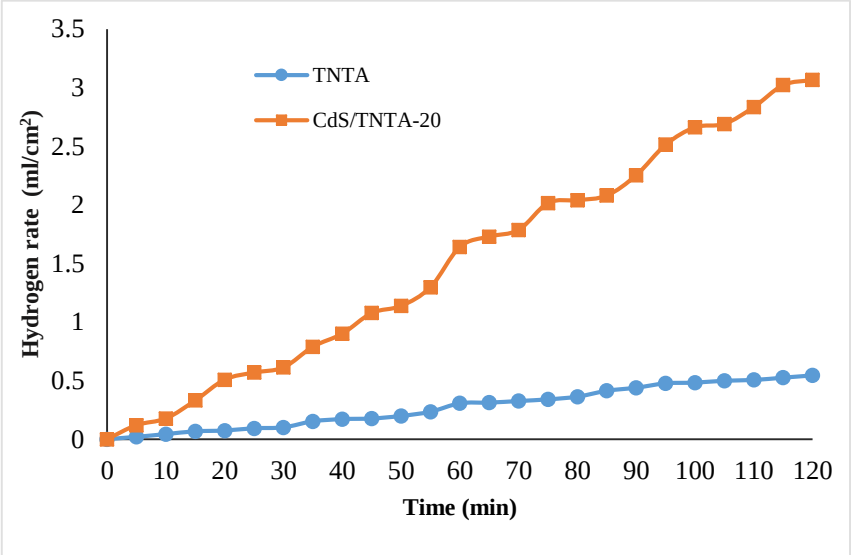


Fig 3.48: Hydrogen rate of electrode

The photocatalytic activity was tested by the water photoelectrolysis. The amount of generated H_2 gas in 2 hours over TNTA and CdS/TNTA electrode, presented in Fig. 3.48.

After 5 minutes under irradiation conditions, generated hydrogen rate on modified TNT electrode (CdS/TNTA-20) averaged 1.5 ml/h/cm^2, which is 5.6 times higher than the one obtained by TNTA (0.24 ml/h/cm^2). The results also showed that, CdS/TNTA-20 electrode is more effectively than the published results of the studies [42, 46, 57]

with hydrogen rate of 1.12 ml/h/cm^2, 30.3 □mol/h/cm^2, 1.53 □mol/h/cm^2, respectively.

CONCLUSIONS

1. Synthesized TNTs by hydrothermal method in alkaline environment, with uniformity size, diameter range from 8-11 nm, with components that anatase phase.
2. Systematic survey of anodized method used to fabricate TNTA and identified suitable working conditions: electrolyte solution containing 0.5% Glycerol, 5% NH_4F with H_2O, temperature, at the voltages 40V, electrolytic time 8 hours, the temperature under the sample processing is 450^{o}C, 3h. The obtained TNTA is uniform tube array structure, length of 3 micron, diameter of 130 nm; with components that anatase phase.
3. Studied modifiing TiO_2 nanotubes (TNTs) powder with various agents including Fe, Cu, Cd, Co, Ni, C, N, Ag, CdS, Pt/rGO and identified agents, which can fabricate a catalysts with higher photochemical activity, has the potential to be used as catalytic to coat on electrodes, applications for water photoelectrolysis process is Pt/rGO and CdS.
4. Successfully synthesized a Pt/rGO/TNTs composite by hydrothermal method. The characteristics result of this composite clearly shows role of Pt and RGO in reducing the recombination of photogeneration electrons/holes pair and increases photocatalytic performance of TNTs.
5. Studied on fabricating electrode by dipping and spin coating methods using TiO_2 sol as a binder on the object are the FTO electrode and TNTs catalyst powder. The results showed that, both methods present good adhesion of the catalyst on the electrode surface. However, dipping method is more limited than spin coating method, FTO electrode diped in a slurry containing catalytic, that can only take a very limited amount of catalytic on electrode surface.
6. Photoactivity evaluated results of catalyst coated electrode in MB photodecomposition reaction showed catalyst Pt/rGO/TNTs coated electrode exhibie higher photochemical activity. This has opened up

application trends of Pt/rGO/TNTs catalyst coated electrode in water photoelectrolysis reaction into hydrogen.

7. Systematically studied on fabrication method CdS doped TNTA electrode and identified: 20 immersion cycles, CdS/TNTA-20 electrode exhibit the best photochemical performence.
8. Surveyed and evaluated the stability of the CdS/TNTA-20 electrode in the water photoelectrolysis reaction. The result shows that in the S^{2-} ion containing electrolyte solution, operations of this electrode are more stable and durable than these in the conventional KOH electrolyte solution. The photocatalytic activity in the photoelectrolysis shows that, the amount of hydrogen generated reached 1.5 ml/h/cm^2, is 5.6 times higher than this of TNTA electrode.

PETITION AND SUGGESTION

The thesis has opened up application trends of Pt/rGO/TNTs catalyst coated electrode via water photoelectrolysis reaction into hydrogen. Under appropriate conditions, the team of reseeachers and PhD candidate proposed the contents for this study as follows:

- Study on improving stability of the Pt/rGO/TNTs catalyst coated electrode in electrolyte environment; optimise the electrode fabrication conditions
- Further study the application of electrodes via water photoelectrolysis reaction into hydrogen.