

Full Paper

Reactor Design Development of Chemical Oxygen Demand Flow System and Its Application

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Abstract- Development of Chemical Oxygen Demand (COD) flow system has been conducted by using TiO₂/Ti electrode. This study aims to develop the flow system portable reactor can be used for determination of COD value as a model design in a river, gutter, and disposal of industrial wastewater. A highly ordered the TiO₂ nanostructure was grown onto Ti plate by anodizing method then calcinated in temperature 500 °C for 1.5 h. The electrochemical test on TiO₂/Ti electrode was performed by using Linear Sweep Voltammetry technique to investigate the high photo-oxidation when ultraviolet (UV) light irradiation. Specifically, the determination of COD value of Sodium Lauryl Sulfate, Hexadecyltrimethylammonium bromide, and 4-oktilfenol poly-ethoxylate surfactants in flow system was applied by using Multi-Pulse Amperometry technique based on Faraday's Law ($Q = \int I_{net} dt$). The excellent sensing of COD value using TiO₂/Ti electrode by flow system showed that SLS, HDTMA-Br, and Triton X-100 surfactants in the concentrations of 1.0 mg/L, 3.0 mg/L, 5.0 mg/L, 7.0 mg/L, and 9.0 mg/L were (1.33 mg/L, 2.92 mg/L, 4.63 mg/L, 6.68 mg/L and 9.43 mg/L); (0.78 mg/L, 3.25 mg/L, 5.21 mg/L, 7.42 mg/L and 9.06 mg/L), and (0.71 mg/L, 3.21 mg/L, 5.05 mg/L, 7.42 mg/L and 8.61 mg/L), respectively.

Keywords- COD sensor, TiO₂/Ti electrode, Photoelectrocatalysis, Surfactants, Flow system

1. INTRODUCTION

Water quality management according to the Ministry of Health in Republic Indonesia the drinking water must be categorized in physical, microbiological, chemical, and radioactive

properties [1,2]. Today, the soil water content has a standard for water consumption in community regarding that the easy to obtain in many regions. However, the pollution level as organic matters were easily found in community area due to the accumulation in aquatic environment [3,4]. Organic matters effect proportionally related with the oxygen demand as well as chemical parameters such as Dissolved Oxygen (DO), Biological Oxygen Demand (BOD), and Chemical Oxygen Demand (COD). These compounds were accumulated in aquatic area by low concentration can be disturbing the oxygen reduction, so still required the detection of water quality [5-7]. The varieties parameters commonly used to organic matters detect by using BOD and COD parameters [8,9].

Nowadays, the COD parameter as standard wastewater pollution was applied in many countries such as Japan and China, is an important parameter in water quality and to determine oxygen demand on organic pollutants [10]. In addition, it is the accurate and faster method for organic pollutants detecting in industries. A principle of COD measurement were the measurement of oxygen demand total which can be consumed by reaction in the aquatic environment [11,12]. It can be determined by using the strong oxidizer agent likes $K_2Cr_2O_7$ as a conventional standard method to determine COD value in wastewater. This method uses acid and silver sulfate solvents as catalyst agents then using the high-temperature preparations so needed a long time to analyze and involve the human toxicity [13-15].

Previously, we have studied of dichromate toxicity effect in conventional method will impact on aquatic environment. An approach to the COD parameter has been investigated by researchers regarding alternative methods to replace the conventional method with the environmental friendly such as electrochemical process [16]. It was considered that the electrochemistry performance can be determining of COD value using titanium dioxide (TiO_2) as oxidation agent [16]. Basically, it was applied on photocatalyst that commonly consists of photon absorb to separate the electron-hole to high ordered oxidation reaction [17,18]. TiO_2 photocatalyst has received many interests to overcome the organic pollutant because of cheaper, simpler, faster, direct measurement, eco-friendly, absolute, and easily for automation [19-21].

Some previous studies, the determination of COD value by electrochemistry method which widely used is a batch system [22-24]. It was considered that not applicable in a real samples area on the river, gutter, and disposal of industrial wastewater. This paper, we have designed of flow system reactor as a model for COD determine in surfactant samples likes Sodium Lauryl Sulfate (SLS), Hexadecyltrimethylammonium bromide (HDTMA-Br), and 4-oktilfenol poly-ethoxylate (Triton X-100). This related to the organic active matters which difficulty for degradation in nature caused of anionic, cationic, or nonionic properties [25,26]. The surfactants can be found in soaps and detergents usually used as a cleaning agent to reduce the surface tension of water and to increase the stability an emulsion system. Furthermore, in low concentration (1 mg/L) surfactant shows that the easy for bubbles

forming and still difficult to degrade caused the stability emulsion and the surface tension system.

Flow system reactor has been studied regarding the COD determination by photoelectrocatalytic system most effective to degrade organic matter that the simultaneous movement of organic compounds to be degrading by TiO₂/Ti electrode. The role of this phenomenon no occur the saturated condition on TiO₂/Ti electrode as well as contributing for COD sensor detection to analyze the level of water pollutions.

2. EXPERIMENTAL

2.1. Design of TiO₂/Ti electrode and flow system reactor

The preparation of Ti (Titanium) plate was done by cutting Ti plate in size of 4.0 cm×1.0 cm. Then, it was sanded by 1200 CC fine sandpaper until clean and shine then washed with the detergent solution, water, and distilled water. Subsequently, Ti plate was immersed (etching) using a mixed solution of HF, HNO₃, and distilled water in the ratio 1:3:6 for two minutes. Anodizing process to obtain the TiO₂/Ti electrode, where the Ti plate inserted in the probe were containing of 0.27 M NH₄F and distilled water with a 98% glycerol. The Ti plate as anode and Cu plate as cathode by connecting a power supply. This anodizing process was carried out for 4 h and calcinated on 500 °C for 1.5 h.

Furthermore, the flow system probe reactor was designed by using cylinder glass in diameter of 1.65 cm, 4.0 cm high, and 7.5 mL volume. In addition, It has been fabricated by three electrodes inserted likes working electrode, Pt as a counter electrode, and Ag/AgCl as a reference electrode while the reactor has been made from plywood coated by aluminum foil. Finalization, it was inserted by 8 pieces the UV lamp (4 Watts) to high performance in TiO₂ photoelectrocatalyst.

2.2. Activity test of TiO₂/Ti electrode

TiO₂ photoelectrochemical activity test by using Linear Sweep Voltammetry (LSV) technique were the 0.1 M NaNO₃ solution added into probe glass. Then, the TiO₂/Ti electrode was connected with a potentiostat (DY2100B) in scan rate of 1×10⁻⁴ V/s and bias potential -1 to 1 volt. The variation of UV light irradiation to investigate the optimization of TiO₂/Ti performance ordered to be used as flow system.

2.3. Measurement of Photocurrent Response to the Surfactants Test Compound

TiO₂/Ti electrode was tested by using the Multi-Pulse Amperometry (MPA) technique with duration of 60 s, and 0.5-volt bias potential with UV light irradiation. The determination

of blanko (0.1 M NaNO₃) as an standard method for I_{blank} variable, meanwhile the surfactants (+0.1 M NaNO₃) as sample to obtain the I_{net} variable.

2.4. Determination of Chemical Oxygen Demand (COD) of the Flow System

Photoelectrochemical system described is capable of stoichiometrically mineralizing surfactants at the working electrode that can be represented as [22];



According to Eq. (1) the number of electrons transferred during the photoelectrocatalysis process can be calculated:

$$n = 4y - 2j + m - 3k - q \quad (2)$$

n value is the electron transfer total when the mineralization surfactants. The next step to determine the number of charge with equation of:

$$Q = \int I_{net} dt = nFCV = (4y - 2j + m - 3k - q)FVC = kC \quad (3)$$

Where:

Q = Coulomb charges

I_{net} = Photocurrent net

n = Number of electrons transfer

V = Volume sample (L)

C = Concentration (mg/L)

Photoelectrocatalytic degradation of surfactants was carried out on TiO₂/Ti thin film by flow system. This process is occur in bulk electrolysis system where the analytes electrolyzed was applied by using Faraday's Law to quantify the COD value by measuring the photocurrent response. The oxygen molecule equivalent with four electrons transferred (on reduction oxygen reaction, O₂ + 4H⁺ → 4e⁻ + 2H₂O), so it can be calculated by the following Eq. 4.

$$COD(mg / L O_2) \frac{Q}{4FV} \times 32,000 = 8000 \times nC = kQ \quad (4)$$

3. RESULTS AND DISCUSSION

3.1. Design of UV reactor based on TiO₂/Ti photoelectrocatalysis

This study has been fabricated the flow system reactor based on UV light optimum intensity caused the high ordered on TiO₂/Ti nanostructure to degrade surfactants sample. Fig. 1A and Fig. 1B shows the flow system reactor with 8 pieces UV lamp by 4 electrical ballasts-assisted to distribute electrical energy. The role of ballasts-assisted to the AC-DC converter, stability electric current, and as a starter to turn on UV lights [27]. This case the electrical circuit has designed by parallel systems in order to efficiency the electrical distribution.

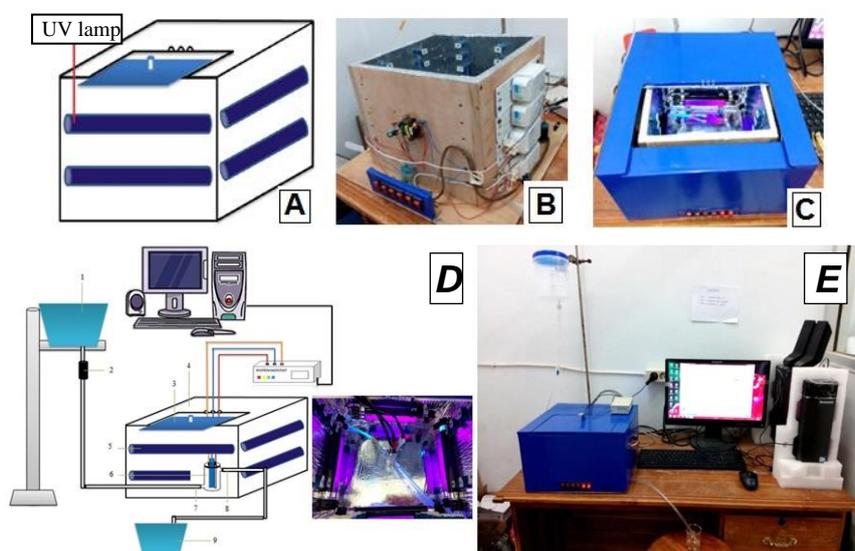


Fig. 1. Design of UV reactor; **(A)** The reactor design; **(B)** The electrical circuit design, and **(C)** The UV reactor system; **(D)** The scheme of flow system reactor; (1) samples container, (2) flow rate regulator, (3) the reactor cover, (4) the box-shaped reactor, (5) UV lights, (6) flow system probe, (7) inlet water, (8) outlet water, (9) the final test solution container; **(E)** The scheme of TiO₂/Ti photoelectrocatalysis flow system reactor

The intensity effect has observed by using TiO₂/Ti electrode in flow system reactor to obtain the high-activity in photoelectrocatalysis system. Variation of UV light irradiation to exhibit the real performance of TiO₂/Ti electrode was activated under UV light because of a band gap of 3.2 eV which active in 388 nm [28]. It was proportional with the photon energy absorption to excite electron-hole which required the high energy.

The TiO₂/Ti electrode has been conducted then tested by flow system reactor with adjusting the UV light intensity using 4 switches. The schematic of flow system reactor using TiO₂/Ti electrode to determine of COD value can be seen in Fig. 1D and 1E. Specifically, the UV lamp intensity variation was investigated based on the photoelectrocatalytic to obtain the

high performance in time and high-activity by flow system. The high improved of TiO_2/Ti electrode activity has studied in LSV technique and the determination of COD flow system by MPA technique.

3.2. Design of TiO_2/Ti electrode and flow system probe

Fig. 2A and 2B shows the flow system probe has been fabricated by a cylinder glass in diameter of 1.65 cm, 4 cm high, and 7.5 mL volume. It has three cover holes consisted as the TiO_2/Ti working electrode, the Pt counter electrode, and the Ag/AgCl reference electrode. Furthermore, the wall probe perforated which function as water inlet/outlet to explain the schematic flow system as determination of COD flow system.

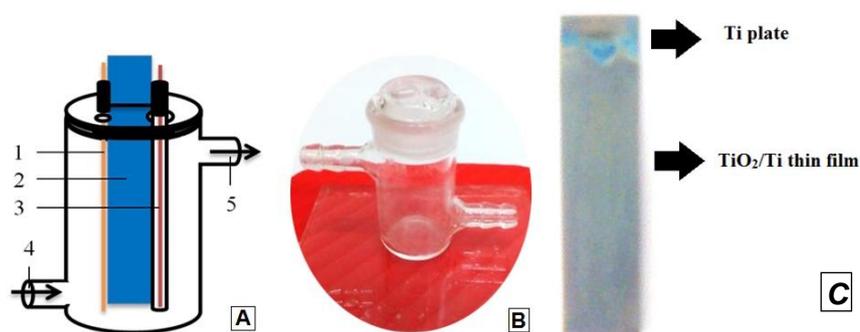


Fig. 2. The design of flow system probe (A) (1) Pt as a counter electrode (2) TiO_2/Ti as a working electrode, (3) Ag/AgCl as a reference electrode, (4) the water inlet, (5) the water outlet; (B) Flow system probe has been designed; (C) TiO_2/Ti thin film electrode

Fig. 2C showed the TiO_2/Ti electrode fabricated from oxidation reaction by an anodizing method. It explained that the electrolysis process was produced the oxide layer on Ti plate. According to Nurdin et al. the anodizing method using bias potential of 25 volts to fabricate the nanotubes structure on Ti plate. It is a standard method for fabricating of electrode based on a semiconductor. At the final stage, the TiO_2 calcinated in temperature $500\text{ }^\circ\text{C}$ for 1.5 h to obtain the anatase crystal which can be applied in photoelectrocatalyst. In addition, this process to evaporate the solvent agent in order to modify TiO_2 crystal [4]. The purpose of utilization of TiO_2 thin-film in photoelectrocatalytic was to overcome water pollution impact on aquatic environment. It was a photo-active caused can absorb the photon energy to produce the oxidation reaction when irradiated by UV light to degrade organic pollutants.

3.3. Effects of UV light intensity against the TiO_2/Ti electrode performance

TiO_2/Ti electrode was tested by UV light intensity in dark condition, 8 Watts, 16 Watts, 24 Watts, and 32 Watts to obtain the variation of photocurrent activity. Fig. 3 can be seen the photocurrent variation when irradiation of UV lights.

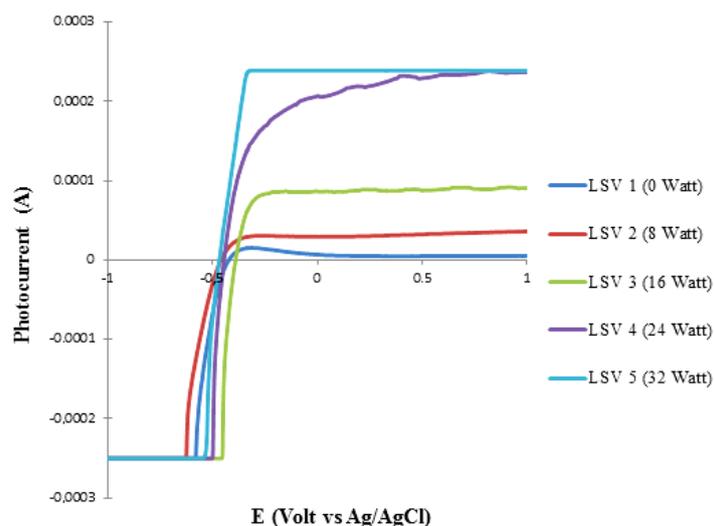


Fig. 3. The variation of LSV graph with UV lights intensity using TiO_2/Ti electrode

Fig. 3 explained that the TiO_2/Ti electrode performance using LSV technique by UV lights variation. It can be seen the existence of voltammograms variation to obtain the photocurrent effects by high UV lights intensity variations. The activity test in dark condition showed that the photocurrent near the zero value was appropriated with Nurdin et al. that the TiO_2 photoelectrocatalyst can not to able in dark condition due to the absence of photon source to separate the electron-hole in TiO_2 semiconductor. This phenomenon has the difference conditions with the UV lights variation intensity to produce of high photocurrent performance [1]. The fifth data (LSV 5: 32 watts) indicated that the high photocurrent activity performance which the simultaneous electron move in TiO_2/Ti electrode so potentiostat overflow condition occupied. It was indicated that can not to use as the determination of COD value in 32 watts.

3.4. Determination of COD flow system on surfactants sample using TiO_2/Ti electrode

The photocurrent illumination was investigated using 24 watts UV light intensity for 60 s by using potentiostat based on standard data (Fig. 3) for high activity of TiO_2/Ti as a working electrode to high ordered the photocurrent response. The 0.5 volt bias potential was used to purpose the optimum mineralization process in flow system reactor. Determination of surfactants photocurrent response was conducted by Multi-Pulse Amperometry (MPA) technique to exhibit the electron transfer on the TiO_2 semiconductor. In addition, It was considered that the electrical current in photoelectrocatalytic performance. Subsequently, the photocurrent net (I_{net}) was calculated by reducing the photocurrent of a sample (I_{sample}) with photocurrent of NaNO_3 (I_{blank}) [22]. The technique of COD determination by flow system design were the surfactants added into probe system, then in the first running off 10 s without

UV light irradiation to obtain the stability of electron transfer, next step the 50 s the illuminated with UV light serve as the electron separation occurs that redox reaction on TiO_2/Ti electrode.

3.4.1. Determination of COD value of SLS anionic surfactant

SLS has classified in surfactant groups and commonly used in domestic and industry. It has been categorized in anionic surfactant which containing of sulfate groups and produces Na^+ as a counter ion. This study, the photocurrent response of SLS has measured by varying concentration were 1.0; 3.0; 5.0; 7.0, and 9.0 mg/L.

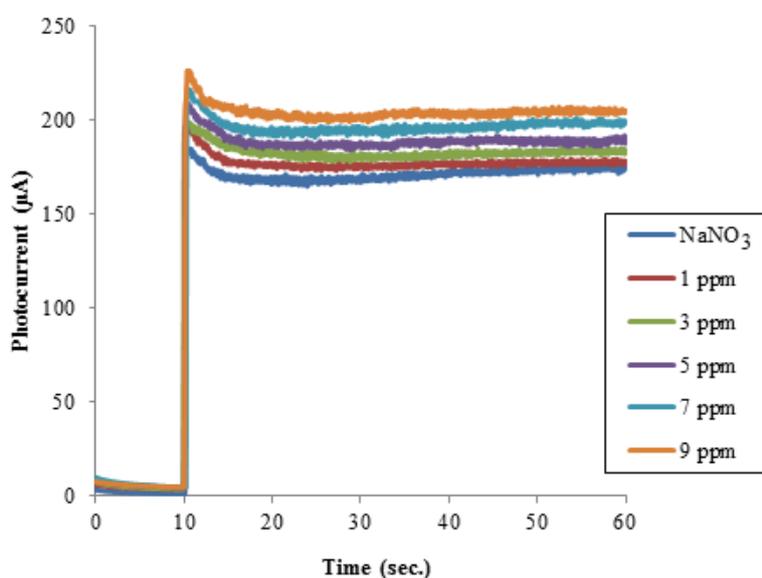


Fig. 4. The photocurrent response on SLS

Fig. 4 showed the photocurrent was proportional with the increase of concentration. According to Maulidiyah et al. the photocurrent response was proportional with the increase of SLS concentration [16]. The high photocurrent response indicated that the high stability performance for degrading surfactant sample and the light intensity in order to separate of electron-hole in TiO_2 semiconductor.

The role of electrolyte solution (0.1 M NaNO_3) was to enhance the conductivity performance in photoelectrocatalytic system. Photocurrent stability by flow system (steady state condition) indicated that the 60 s process to replace of samples on bulk by flow system due to increase current.

Table 1. The I_{net} response of SLS

Concentration (mg/L)	I_{sample} (μA)	I_{blank} (μA)	dt (s)	I_{net} (μA)	Q (μC)
1.0	197.31	184.728	60	12.582	754.92
3.0	202.9704	184.728	60	18.2424	1094.544
5.0	209.1052	184.728	60	24.3772	1462.632
7.0	216.3817	184.728	60	31.6537	1899.222
9.0	226.2172	184.728	60	41.4892	2489.352

3.4.2. Photoelectrochemical response to the HDTMA-Br cationic surfactant

HDTMA-Br has an aliphatic group of 4 carbonic atoms. The photoelectrochemical response of the HDTMA-Br was shown in Fig. 5.

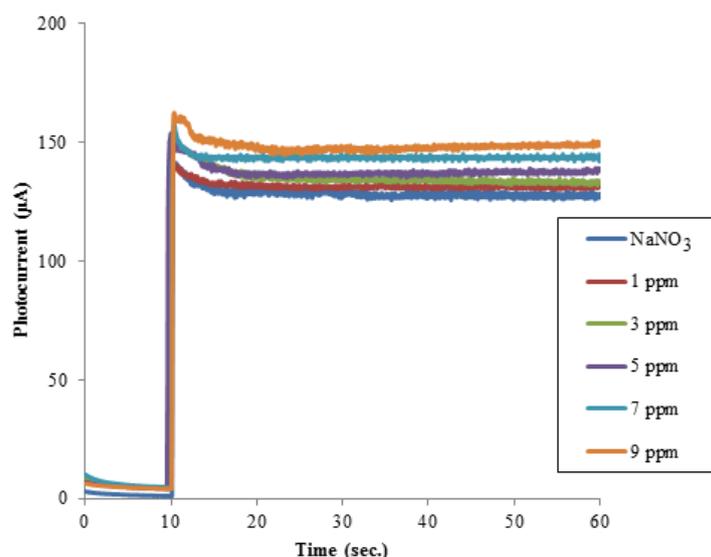
**Fig. 5.** The photocurrent response on HDTMA-Br

Fig. 5 exhibited the amperomogram of HDTMA-Br was different between the cationic and anionic surfactants. This condition has obtained that the low photocurrent value caused the decrease of water splitting to produce electron transfer on bulk system. In addition, the ammonium ion can influence the reference electrode. Based on Zhybak et al. that the reference electrode not working due to containing complex ions such as ammonium ion [29].

Table 2. The I_{net} response of HDTMA-Br

Concentration (mg/L)	I_{sample} (μA)	I_{blank} (μA)	dt (s)	I_{net} (μA)	Q (μC)
1.0	143.783	141.4821	60	2.3009	138.054
3.0	149.3421	141.4821	60	7.86	471.6
5.0	153.7558	141.4821	60	12.2737	736.422
7.0	157.1161	141.4821	60	15.634	938.04
9.0	162.428	141.4821	60	20.9459	1256.754

Table 2 can be seen the photocurrent response was proportional with the increasing of HDTMA-Br concentrations. This condition indicated that the high-linearity compared with the other samples.

3.4.3. Photoelectrocatalysis response to Triton X-100 nonionic surfactant

In this study, the Triton X-100 as a nonionic surfactant commonly known as 4-octylphenol polyethoxylate have the common of photocurrent response with the other surfactants type. In addition, this treatment to observe the photoelectrocatalysis response also as a comparative compounds. The photocurrent response has conducted by using potentiostat were can be seen in Fig. 6.

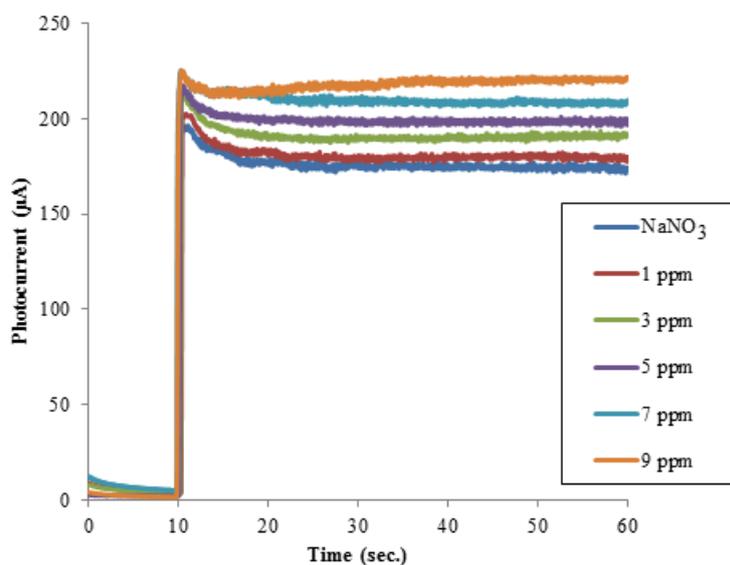
**Fig. 6.** The photocurrent response of Triton X-100

Fig. 6 showed the degradation process of non-ionic surfactant can be able degraded by TiO_2/Ti electrode. The photocurrent graph was separated in the each Triton X-100

concentrations. According to da Silva et al. that the 4-nonylphenol ethoxylate (nonionic) indicated that the perfectly of oxidation degradation using the photoelectrocatalysis system [30].

This study was used the nonionic surfactant which contained the longest carbon chain compared with the other surfactants. It has a reduplication of ethoxylate groups was easily attacked by radical OH to degrade the Triton X-100. The quickly of degradation process was influenced by photocurrent effect in amperomogram profile. In other hands, the existence of benzene groups also to difficulty for spontaneous degradation process.

Table 3. The I_{net} response of Triton X-100

Concentration (mg/L)	I_{sample} (μA)	I_{blank} (μA)	dt (s)	I_{net} (μA)	Q (μC)
1.0	202.561	195.9743	60	6.5867	395.202
3.0	210.9972	195.9743	60	15.0229	901.374
5.0	217.2271	195.9743	60	21.2528	1275.168
7.0	225.2536	195.9743	60	29.2793	1756.758
9.0	229.2536	195.9743	60	33.2793	1996.758

Table 3 showed that the relevant with the high-concentrations. This condition can be observed that the correlation charge between non-ionic surfactants concentration. This result can be applied in flow system at the environment.

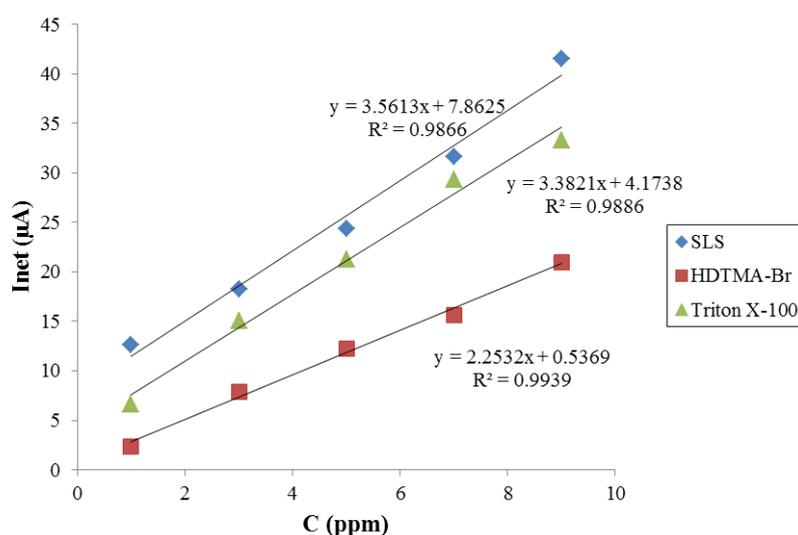


Fig. 7. The relation between I_{net} and concentration of surfactants

3.5. Response of photocurrent net (I_{net}) to the all surfactants types

In the previous discussion has been shown the relation between I_{net} and the concentration in each surfactants sample. According to the Faraday's Law that the Q value proportional with the concentration, $Q=nFVC$ [8]. It has observed the different values in a slope curve to explain the linearity and sensitivity on TiO_2/Ti electrode against the sample. The high photocurrent response exhibits the high sensitivity, linearity, and accuracy.

Fig. 7 showed the relation I_{net} between concentrations of surfactants. According to researchers to degrade the surfactants using TiO_2 photoelectrocatalyst explained that the surfactants effect high performance in the degradation process. Fig. 7 exhibited the different slope of each surfactant compounds. The anionic surfactants effect have the high I_{net} slope compared with other surfactants type were the degradation process in surfactant high-performance then non-ionic and cationic surfactants.

This condition has ordered the positive charge on TiO_2/Ti electrode indicated the working electrode in positive charge then the anionic surfactants can be easily to absorb on TiO_2 surface. The high-absorption process was effectiveness and proportional with the photocurrent response between concentration exhibit the high value on the slope curve.

HDTMA-Br has indicated the smallest slope caused is difficult to degrade by TiO_2 photoelectrocatalyst. The effect of positive charge on TiO_2/Ti electrode and HDTMA-Br as cationic surfactant involve the separation charge, partially the reaction process was degraded in bulk system. This reaction mechanism exhibited the attack effect of an electron in Pt electrode. Meanwhile, the hole was initiated oxidation reaction not occur [31].

3.6. Determination of COD value by flow system

The determination of COD value theoretic has been investigated to exhibit the concentration effect in the sample due to the electron transfer total. It was obtained the COD value theoretic was increasing along with the surfactant concentrations.

Table 5. The COD value by flow system for all surfactant types

C (mg/L)	COD theoretical (mg/L O ₂)			COD flow system (mg/L O ₂)			COD diffenrent (mg/L O ₂)		
	SLS	HDTMA- Br	Triton X-100	SLS	HDTMA-Br	Triton X-100	SLS	HDTMA-Br	Triton X-100
1.0	1.808	2.504	2.176	1.33	0.78	0.71	0.478	1.724	1.466
3.0	5.416	7.512	6.536	2.92	3.25	3.21	2.496	4.262	3.326
5.0	9.04	12.56	10.88	4.63	5.21	5.05	4.41	7.35	5.83
7.0	12.64	17.52	15.28	6.68	7.42	7.42	5.96	10.1	7.86
9.0	16.24	22.56	19.6	9.43	9.06	8.61	6.81	13.5	10.99

This phenomenon the highest ordered of organic compounds degradation was proportional with the COD value and given the information of surfactants degraded perfectly by TiO₂ photoelectrocatalyst.

The increasing of solution concentration was proportional by COD value on each test compounds. It could be seen that the SLS has highest of COD value in 1 mg/L for 1.33 mg/L O₂. However, It occurred decreasing of COD value on the concentration of 3 mg/L, 5 mg/L, and 7 mg/L compared with the other of surfactant types was 2.92; 4.63 and 6.68 mg/L O₂. In the 9.0 mg/L concentration, the COD value was increasing for 9.34 mg/L O₂. This showed that each concentration of the organic compounds has the different degradation process in the photoelectrocatalysis flow system caused the saturated of TiO₂/Ti surface in all systems.

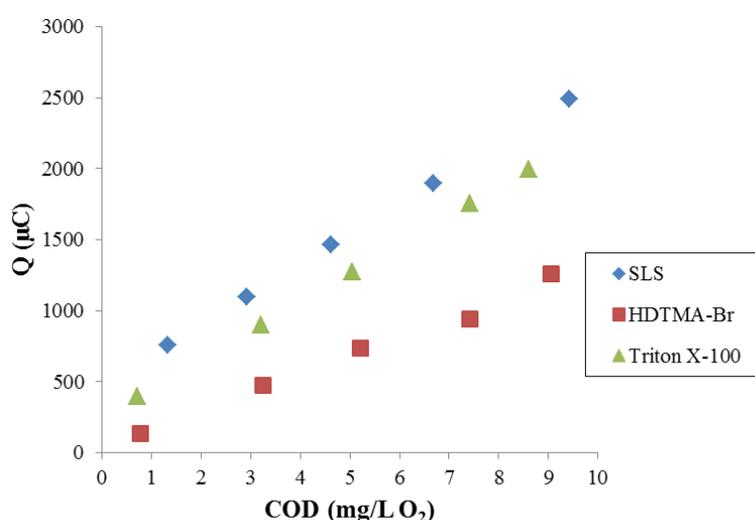


Fig. 8. Graphic of charge (Q) relation and the COD value in flow system

Fig. 8 showed that the anionic surfactant (SLS) was the highest COD value between the other types of surfactants in 9.43 mg/L O₂. This result showed that the SLS compound has experienced the better degradation process. Supported by the negative charge on SLS that was strongly absorbed on the TiO₂ surface as a positively charged caused the electron transfer would easily happen by flow system.

4. CONCLUSION

The UV light intensity was used to determine of TiO₂/Ti electrode optimum activities that the 24 watts power increased the intensity to degrade surfactants sample. The determination of COD value by flow system reactor using TiO₂/Ti electrode showed that SLS, HDTMA-Br, and Triton X-100 compounds in the concentration of 1.0 mg/L, 3.0 mg/L, 5.0 mg/L, 7.0 mg/L, and 9.0 mg/L were (1.33 mg/L, 2.92 mg/L, 4.63 mg/L, 6.68 mg/L and 9.43 mg/L);

(0.78 mg/L, 3.25 mg/L, 5.21 mg/L, 7.42 mg/L and 9.06 mg/L), and (0.71 mg/L, 3.21 mg/L, 5.05 mg/L, 7.42 mg/L, and 8.61 mg/L), respectively.

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