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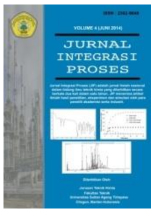

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THE ANALYSIS OF POLLUTANT PARAMETERS IN TOFU WASTEWATER AFTER BEING TREATED BY CONTACT GLOW DISCHARGE ELECTROLYSIS

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Abstract

There are many Small, Micro, and Medium Enterprises engaged in tofu processing in Indonesia. Some of them still dumped their waste directly into the river, causing the stream to become turbid, had a bad odor, and many aquatic organisms in the water died. Tofu wastewater contained high pollutant parameters, such as COD, BOD, TOC, and TSS; therefore, we need an effective wastewater treatment that could reduce the level of these parameters below the government's standards. Contact Glow Discharge Electrolysis (CGDE) was used to treat the wastewater. Besides these four parameters, pH solutions were also measured at several CGDE process voltage variations (such as 650 V, 700 V, and 750 V). The content of compounds contained in the initial tofu wastewater and the waste that had been treated by using CGDE were analyzed. From this study, it was known that the higher the voltage applied, the more acidic the solution would become due to hydrogen peroxide emersion, which is a weak acid. In the same operating condition, the percentage of TOC degradation was lower than that of COD degradation. The voltage of 750 V gave the lowest COD and TOC values, respectively 446.6 mg/L and 320 mg/L. Also, the best voltage that could reduce BOD and TSS degradation by 37% and by 80.2% respectively, was 650 V. Although CGDE process was able to decrease COD, BOD, TOC, and TSS, the decrease of these pollutant parameters still could not reach the standards set by the government, except for TSS. Besides having the ability to reduce pollutant parameters, it is also powerful in degrading complex compounds in tofu wastewater into compounds with simpler molecular structures, such as Tris (2,4-di-tert-butyl phenyl) phosphate and eicosane.

Keywords*:* BOD; COD; Compound analysis; TOC; TSS

1. INTRODUCTION

Most tofu making processes produce liquid waste that has high pH, BOD, COD, and Total Suspended Solid (TSS). The high level of the pollutant parameters was caused by waste, which has high acid content and bad odor (Kaswinarni, 2007). Besides, the contamination level in the waste was usually caused by complex organic materials present in the tofu wastewater. Fresh tofu wastewater still contains high pollution parameters that could be seen in Table 1.

Table 1. Pollutant parameters in fresh tofu wastewater

Pollutant parameters	Value
pH	3.9*
COD (mg/L)	7771.3*
BOD (mg/L)	2547.5
TSS (mg/L)	840.5*

* (Pangestika et al., 2018a)

Contact Glow Discharge Electrolysis (CGDE) is a process to degrade organic pollutants in wastewater by using plasma (Sharma et al., 2015; Saksono et al., 2013). The process of organic pollutant degradation by CGDE was started by charged species forming on the plasma around the anode. Hydroxyl radicals would be produced through the homolytic dissociation of water molecules through the formation of plasma that comes in contact with the surface of the solution (Tomizawa & Tezuka, 2007).

Hydroxyl radicals are the most potent species among all radicals and species formed during the CGDE process. They are the main species that would attack organic materials, as can be seen in Equation 1 (Yang et al., 2013). Hydroxyl radicals are oxidizing agents of the tracks, which could damage the benzene or naphthalene ring in organic pollutants (Ren et al., 2014).

$$\text{Organic} + OH\bullet \rightarrow \text{Products} \quad (1)$$

According to Wang & Jiang (2009), if hydroxyl radicals attack pollutants in the waste, carbon dioxide gas and water vapor will form as a product, according to Equation 2,

$$OH\bullet + \text{pollutant} \rightarrow CO_2 + H_2 \quad (2)$$

One hydroxyl radical could also combine with other hydroxyl radicals to form hydrogen peroxide. The formed hydrogen peroxide can regenerate hydroxyl radicals again through additional Fenton reagents to increase the rate of degradation of organic pollutants (Tomizawa & Tezuka, 2007).

2. MATERIALS AND METHODS

The research was conducted from August 2017 until February 2018 at the Intensification Laboratory of Universitas Indonesia.

This research studied a few things, such as compound analysis in tofu wastewater at different time variations and the relationship between voltage variation to pH, COD, BOD, TOC, and TSS analysis. The materials used in this research were tofu wastewater that had been pre-treated by the coagulation-flocculation method (Pangestika et al., 2018a), Na_2SO_4, and $FeSO_4.7H_2O$. Equipment that was used in this study was the experimental setup for the CGDE process (could be seen in Figure 1), pH meter, UV-Vis Spectrophotometer, TSS meter, and GC-MS.

2.1 Analysis of Compound in Tofu Wastewater at Several Time Variation

As much as 1.5 L of tofu wastewater and Na_2SO_4 0.02 mol/L waste was put into the CGDE reactor. Ion Fe^{2+} in $FeSO_4.7H_2O$ 40 mg/L was mixed into the reactor as a catalyst. The anode was immersed 20 mm from the surface boundary. The air pump associated with the bubbler was turned on at a high flow rate. The voltage regulator was set from 50 V and was increased gradually to 750 V. This voltage was chosen because in this variation the amount of hydrogen peroxide formed was greater (Pangestika et al., 2018b) and the percentage of COD degradation in liquid tofu waste was very high than other variations. At the voltage, CGDE was carried out for 30 minutes, 60 minutes, and 120 minutes. Then, the waste sample was taken, and the compound was measured by using GC-MS at the Indonesia National Police Centre.

2.2 The Relationship between Voltage Variation to pH, COD, BOD, TOC, and TSS Analysis

As much as 1.5 L of tofu wastewater and Na2SO4 0.02 mol/L waste was put into the CGDE reactor. Ion Fe2+ in FeSO4.7H2O 40 mg/L was mixed into the reactor as a catalyst. The anode was immersed 20 mm from the surface boundary. The air pump associated with the bubbler was turned on at a high flow rate. The voltage regulator was set from 50 V and was increased gradually to 650 V, 700 V, and 750 V. At each voltage, CGDE was carried out for 120 minutes. Variation of 0 V was conducted before CGDE took place. Then, the sample of wastewater was taken to measure pH, COD, BOD, TOC, and TSS for every voltage variation. COD was measured by using SNI 6989.2:2009, BOD was measured by using SNI 06-6989-72-2009, TOC was measured by using APHA 5310 B, TSS was measured by using SNI 06-6989-3-2004.

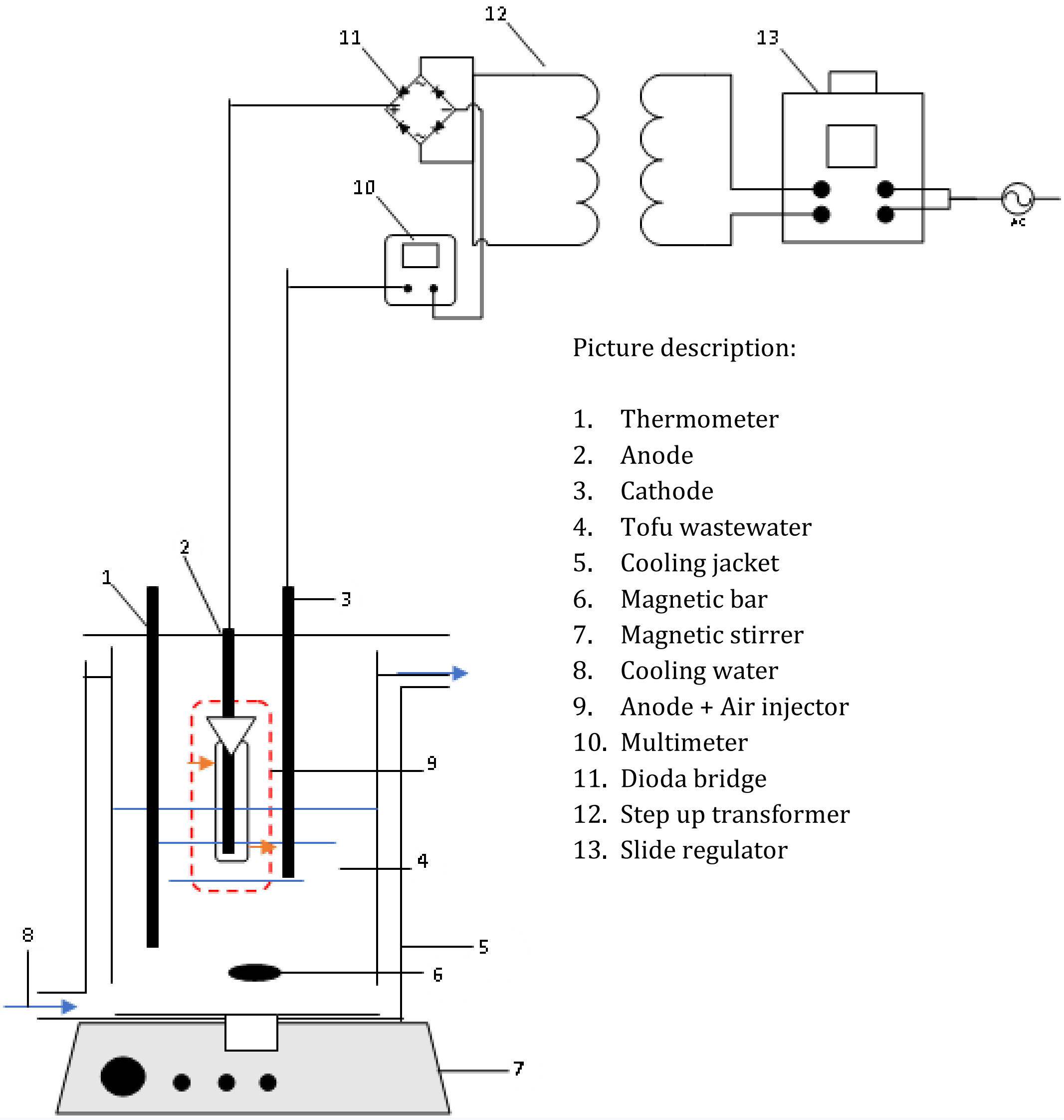

Figure 1. The experimental setup for CGDE process (Pangestika et al., 2018b)

3. RESULTS AND DISCUSSION

Tofu wastewater used in this research was wastewater that had been pre-treated by the coagulation-flocculation method. The pollution parameters before the CGDE process could be seen in Table 2.

Table 2. Pollutant parameters in tofu wastewater that had been pre-treated by coagulation-flocculation method

Pollutant parameters	Value
pH	3.8
COD (mg/L)	1766.6
BOD (mg/L)	520.5
TOC (mg/L)	580
TSS (mg/L)	484

3.1 Analysis of Compound in Tofu Wastewater at Several Time Variation

The most abundant compounds in tofu wastewater are ((3S, 3aR, 6R, 8aS) -7.7-Dimethyl-8-methyleneoctahydro-1H-3a, 6-methanoazulen-3 -yl) methanol. As shown in Table 3, compounds included in the methanol group existed in the tofu wastewater as much as 11.47%.

According to Mazzocchi et al. (1973), radical species were found in methanol compounds, which were included: H•, CH_3•, dan CH_2OH•. This radical reaction could be seen in Equations 3–7,

$$H\bullet + CH_3OH \rightarrow CH_2OH\bullet + H_2 \quad (3)$$

$$CH_3\bullet + CH_3OH \rightarrow CH2OH\bullet + CH_4 \quad (4)$$

$$2CH_2OH\bullet \rightarrow CH_2OH - CH_2OH \quad (5)$$

$$H\bullet + H\bullet \rightarrow H_2 \quad (6)$$

$$CH_3\bullet + CH_3\bullet \rightarrow C_2H_6 \quad (7)$$

From the five reactions above, it could be seen that radical species could degrade methanol compounds into saturated compounds, such as those found in the alkane group. Hydroxyl radicals were also produced in the CGDE process. The reaction of methanol with hydroxyl radicals can be seen in Equation 8 (Buxton et al., 1988),

$$OH\bullet + CH_3OH \rightarrow CH_2OH\bullet + H_2O \quad (8)$$

Table 3. Compound analysis in tofu wastewater before being treated by CGDE

No	Compounds	Area (%)
1	((3S,3aR,6R,8aS)-7,7-Dimethyl-8-methyleneoctahydro-1H-3a,6-methanoazulen-3-yl) methanol	11.47
2	Siklohexane, (4,4-dimethyl-1-methylene-2-pentinil)-	7.97
3	4.beta.H,5.alpha.-Eremophila-1(10),11-diene	7.48
4	(1R,4aS,8aR)-1-Isopropyl-4,7-dimethyl-1,2,4a,5,6,8a-hexahydronaptalen	6.86
5	4-[2',6'-Dimethoxy-4'-methylphenyl]but-3-en-2-one	6.14
6	(8R,8aS)-8,8a-Dimethyl-2-(propan-2-ylidene)-1,2,3,7,8,8a-hexahydronaphthalena	5.88
7	(3S,3aS,5R,6S,7aS)-3,6,7,7-Tetramethyloctahydro-3a,6-ethanoinden-5-o	5.29
8	(3S,3aS,6R,8aS)-3,7,7-Trimethyl-8-methylenoctahidro-1H-3a,6-metanoazulena	4.71
9	(4R,4aR)-4,4a-Dimethyl-6-(prop-1-en-2-yl)-1,2,3,4,4a,7-hexahydronaphthalen	4.46
10	(5R,10R)-6,10-Dimethyl-2-(propan-2-ylidene)spiro[4.5]dec-6-en-8-one	4.31
	Total Area (%)	64.57

If the concentration of methanol in the wastewater has begun to decrease, species $CH_2OH\bullet$ will react with oxygen so that it could form into other radical species to finally obtain the final product in the form of oxygen, methanal, and hydrogen peroxide, according to Equation 9 and Equation 10 (Wang et al., 2008),

$$CH_2OH\bullet + O_2 \rightarrow CH_2OHO_2\bullet \quad (9)$$

$$2CH_2OHO_2\bullet \rightarrow 2HCHO + H_2O_2 + O_2 \quad (10)$$

From the degradation mechanism above, it could be seen that the amount of hydrogen peroxide increased due to the CGDE process.

CGDE process was proven to be able to break the bonds in complex compounds to become simpler. After thirty minutes of the CGDE process, there were many alkane compounds in tofu wastewater, included: eicosane, octadecane, hexatriacontane, and 8-methyl-heptadecane. Table 4 shows that eicosane was the highest amount of compound after thirty minutes of CGDE, which was 37.34%. The eicosane chemical formula is $C_{20}H_{42}$.

At 750 V, a large and stable plasma would be produced. The large plasma would cause the solution temperature to be higher (Mazzocchi et al., 1973). It indicated the formation of unsaturated hydrocarbon compounds, such as diene compounds found in wastewater that has been electrolyzed for thirty minutes.

Table 4. Compound analysis in tofu wastewater after being treated by CGDE for 30 minutes

No	Compounds	Area (%)
1	Eicosane	37.34
2	Octadecane	11.47
3	Hexatriacontane	6.83
4	8-methyl-heptadecane	4.67
5	7,9-di-tert-butyl-1-oxaspiro [4,5] deca-6,9-diena-2,8-dione	2.07
	Total Area (%)	62.38

The quantity of the compound 7,9-di-tert-butyl-1-oxaspiro [4,5] deca-6,9-diene-2,8-dione could be reduced by 24% from the thirtieth minutes to the sixtieth minute of CGDE. It could be seen in Table 5 that a new compound appeared after sixty minutes of CGDE, Tris (2,4-di-tert-butylphenyl) phosphate. The phosphate compound presented in the wastewater as much as 23.03%. As with the phosphate compound, the presence of eicosane compounds at the sixtieth minute of CGDE decreased significantly to 1.41% from 37.34% at the thirtieth minute of CGDE. Table 5 also shows that the amount of 7.9-di-tert-butyl-1-oxaspiro [4,5] deca-6,9-diene-2,8-dione decreased from 2.07% to 1.58%.

Table 5. Compound analysis in tofu wastewater after being treated by CGDE for 60 minutes

No	Compounds	Area (%)
1	Tris (2,4-di-tert-butylphenyl) phosphate	23.03
2	Benzena, 1-(1,5-dimethyl-4-heksenil)-4-methyl	2.11
3	R-1-methyl-4-(6-methylhept-5-en-2-yl) siklohexa-1,4-diena	1.80
4	7,9-di-tert-butyl-1-oxaspiro [4,5] deca-6,9-diene-2,8-diona	1.58
5	Eicosane	1.41
	Total Area (%)	29.93

There was no new compound in the wastewater after the CGDE process for 120 minutes. After 120 minutes, the tris compound (2,4-di-tert-butyl phenyl) phosphate could be half reduced compared to the sixtieth minute. It could be seen in Table 6 that the phosphate compound was found as the most compound after the CGDE process for 120 minutes, with an amount of 10.24%. The percentage of 7,9-di-tert-butyl-1-oxaspiro [4,5] deca-6,9-diene-2,8-dione in the waste also decreased to 0.99%. The octadecane compound was reduced dramatically from 11.47% to 0.31% after 120 minutes CGDE.

The broken carbon chain that was successfully degraded could combine to form eicosane compounds because the number of eicosane compounds at the last minute of CGDE increased to 9.28%, as shown in Table 6. CGDE process was able to degrade complex compounds in tofu wastewater into compounds with simple molecular structures.

Table 6. The results compound analysis in tofu wastewater after being treated by CGDE for 120 minutes

No	Compounds	Area (%)
1	Tris (2,4-di-tert-butyl phenyl) phosphate	10.24
2	Eicosane	9.28
3	7,9-di-tert-butyl-1-oxaspiro [4,5] deca-6,9-diena-2,8-diona	0.99
4	Octadecane	0.31
	Total Area (%)	20.82

3.2 pH Analysis

Tofu wastewater that had not been treated was acidic because it had a low pH, which was 3.8. The acidity of tofu wastewater was due to acetic acid that used to agglomerate soy in making tofu. This process was done due to the higher amount of hydrogen peroxide that was occurred during the CGDE process (Permatasari et al., 2018). Figure 2 shows that the higher the voltage during CGDE, the more acidic the solution would become. The decreasing pH of this solution was due to hydrogen peroxide, which is a weak acid that was formed more at a higher voltage.

The emersion of hydrogen peroxide in the waste made pH of tofu wastewater decreased. This value could not fulfill the Standard by Minister of the Environment Number 15 in 2008 about wastewater criteria that is safe for the environment, which is approximately 6-9. Although hydrogen peroxide brought down the pH of the wastewater, it was an active species that determined the formation of hydroxyl radicals (Jinzhang et al., 2008). Hydroxyl radical was one of the reactive species produced in the plasma electrolysis process. If the hydroxyl radicals formed by plasma electrolysis were more numerous, the quantity of hydrogen peroxide compounds that were presented in the solution would undoubtedly be even more significant (Jinzhang et al., 2007).

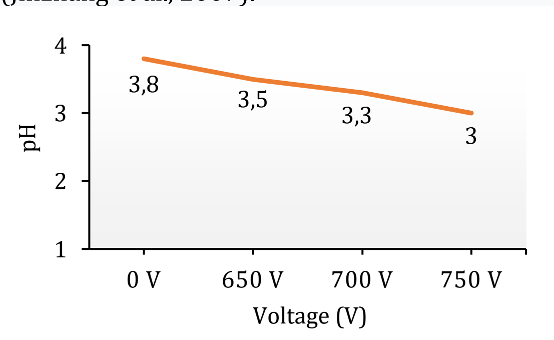

Figure 2. The effect of voltage during the CGDE process to pH of the solution (2 cm anode depth, COD concentration of 2000 mg/L waste, 0.02 M Na_2SO_4, Fe^{2+} 60 mg/L ions, and temperature of 50°C).

3.3 COD and TOC Analysis

According to Wang et al. (2008), COD and TOC were important indicators of water contamination. COD is the chemical oxygen demand needed to oxidize organic compounds in water, while TOC is the amount of carbon contained in organic compounds in waste. COD of tofu wastewater would decrease after being treated with CGDE.

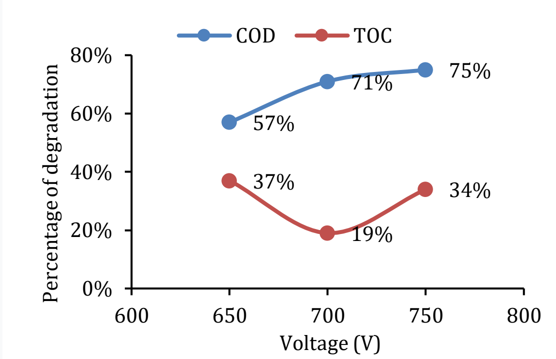

Figure 3. Percentage of COD and TOC degradation in tofu wastewater that has been treated at several variations of CGDE voltage (anode depth of 2 cm, Na_2SO_4 0.02 M, Fe^{2+} 60 mg/L ions, and temperature of 50°C)

Figure 3 describes that the higher the voltage used in the process, the lower COD and TOC in tofu wastewater. Based on Figure 4, a voltage of 750 V gave the lowest COD and TOC values, respectively 446.6 mg/L and 320 mg/L. TOC levels in tofu wastewater were more difficult to reduce than COD levels. It was proved by the more significant percentage of COD degradation, by 75%, than the percentage of TOC degradation at 750 V voltage, which was as much as 45%. This was in accordance with research conducted by Wang et al. (2008) that the percentage of TOC degradation was lower than the percentage of COD degradation in the same operating conditions.

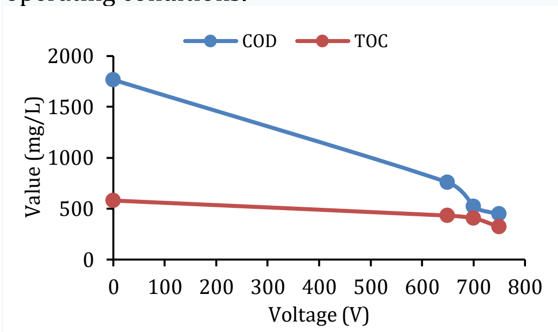

Figure 4. Comparation of COD and TOC before and after treated by using CGDE

3.4 BOD Analysis

Biological oxygen demand, or commonly abbreviated as BOD, is the amount of oxygen needed by microorganisms to break down or break down organic material in tofu wastewater. It was shown in Figure 5 that the most considerable decline in BOD was obtained using a voltage of 650 V during the CGDE process, which was as much as 37%. After the CGDE process, the BOD value in tofu wastewater was still quite high. It might have occurred because the number of organic substrates in wastewater was not being reduced, so the amount of oxygen needed by microorganisms to break down organic matter in wastewater was still quite high.

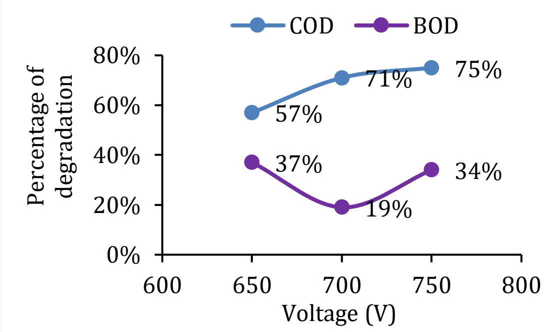

Figure 5. Percentage of COD and BOD degradation in tofu wastewater that has been treated at several variations of CGDE voltage (anode depth of 2 cm, Na_2SO_4 0.02 M, Fe^{2+} 60 mg/L ions, and temperature of 50°C)

Table 7. Ratio of BOD and COD before and after treated by using CGDE

Parameter	Voltage			
	0 V	650 V	0 V	750 V
COD (mg/L)	1766.6	757.3	518.6	446.6
BOD (mg/L)	520.5	326.5	419.5	344.5
BOD/COD	0.29	0.43	0.81	0.77

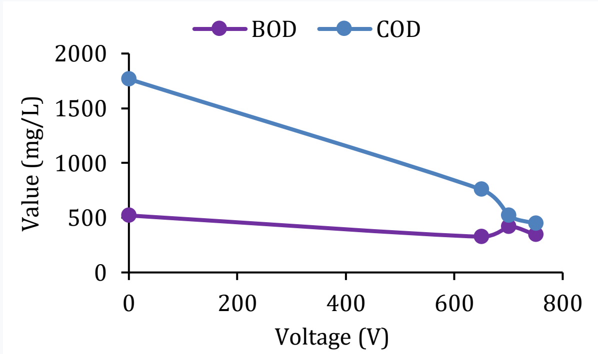

Figure 6. Comparation of BOD and COD before and after treated by using CGDE

According to the data in Table 7, the BOD/COD ratio of tofu wastewater before being processed was 0.29. After being treated by CGDE at a voltage of 650 V, the BOD/COD ratio of waste increased to 0.43. When the CGDE voltage was increased to 700 V and 750 V, the BOD/COD ratio went up to 0.81 and 0.77, respectively. The increase in the BOD/COD ratio indicated that through the CGDE process, organic contaminants in tofu wastewater became easier to degrade biologically, so further chemical treatment was not necessary to reduce the number of contaminants in the wastewater. This was also supported by Romli and Suprihatin (2009), which stated that if the BOD/COD ratio of wastewater was more than 0.4, the materials in wastewater could be easily degraded biologically. It means that it was not necessary to set the voltage above 650 V because the BOD/COD ratio would increase above 0.4.

Overall, Figure 6 shows that the higher the voltage, the more decrease the BOD and COD value. Even there were declines in COD and BOD levels after CGDE, these two values still exceeded the quality standards set by the Minister of Environment Regulation Number 15 in 2008 concerning to tofu wastewater. The quality standard set for COD and BOD levels in tofu wastewater was 300 mg/L and 150 mg/L.

3.5 TSS Analysis

TSS is a total solid retained by a 1.2 μm filter. Figure 7 shows that the TSS levels in treated wastewater with 650 V voltage and 700 V voltage had almost the same value, which was 96 mg/L and 94 mg/L. As with COD and TOC, the level of TSS increased when the voltage used was higher. The rise in TSS value that occurred at 750 V was probably due to the higher voltage, so the anode became more easily eroded. The eroded anode might also increase the number of total solids in wastewater.

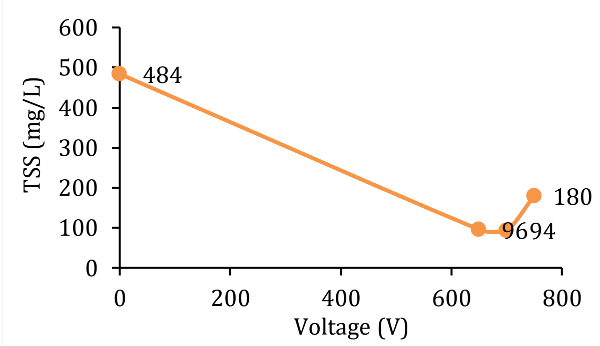

Figure 7. Comparation of TSS before and after treated by using CGDE

Being different from COD and BOD, all TSS levels in wastewater treated with every variation of the CGDE voltage were below the quality standard set by the government, which was 200 mg/L.

4. CONCLUSION

Contact glow discharge electrolysis (CGDE) is an effective method to reduce COD, TOC, BOD, and TSS in tofu wastewater. Although pH, COD, BOD, and TOC still not meet the Standards determined by Minister of Environment, CGDE could lower COD as much as 71%, BOD up to 37%, TOC up to 45%, and TSS as much as 81%. The lower pH of tofu wastewater that had been treated by CGDE made it did not fulfill the standard determined by the Minister of Environment. The best reduction for COD and TOC was obtained by using a voltage of 750 V, which were 446.6 mg/L and 320 mg/L while BOD and TSS were reduced maximally by applying a voltage of 650 V, which were 326.5 mg/L and 96 mg/L. Although CGDE was a potential method for wastewater treatment, the only parameter that fulfilled the standard by the Minister of Environment was TSS.

Besides, CGDE could also degrade complex compounds into simpler compounds, such as Tris (2,4-di-tert-butyl phenyl) phosphate and eicosane, they were the most significant compounds that were found in tofu wastewater after being treated by CGDE for 120 minutes.

5. ACKNOWLEDGEMENT

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