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The Treatment of the Waste Water of Urea Fertilizer Plant with a Combined Process of Advanced Oxidation and Microalga *Chlorella pyrenoidosa*, *Nannochloropsis* sp. and *Pseudomonas fluorescens* Bacteria

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The activity of urea fertilizer industry with potential environmental pollution is the activity of wastewater waters. The main by product of the fertilizer urea containing mostly liquid ammonia. According to ministry of Environmental Affairs No. 122 of the Decree of the Governor of South Sumatra of the year 2005, the maximum pollution load for industry is liquid ammonia level of 0.75 kg/ton (50 of 6.0 – 9.0. The treatment of the wastewater of urea is done by means of combining chemical and biological technology of wastewater treatment by advanced using strong oxidizing agents. The process of combined with or followed by biological processes organisms such as microglae *Chlorella pyrenoidosa*, *Nannochloropsis* sp. And the *Pseudomonas fluorescens* bacteria. The study on the treatment of the wastewater of the urea using Fenton reagent and advanced oxidation shows: the capacity to degrade NH_3 is 95% and ratio of 1:10 and the use *Pseudomonas fluorescens* and nitrite as much as 92.63% microalgae as much as 99% and *Nannochloropsis* sp. as

Advanced Oxidation Process (AOP), *Chlorella* *Nannochloropsis* sp, *Pseudomonas fluorescens*

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I. INTRODUCTION

THERE are six urea fertilizer plants in Indonesia whose wastewater is characterized with high levels of urea and ammonia-nitrogen. The treatment of wastewater with high levels of urea and ammonia-nitrogen is one of the problems faced by urea fertilizer plants in Indonesia. Although the waste water of urea fertilizer plants is not considered as hazardous materials compound, it may cause serious damage to the ecosystem of water bodies. Industrial activities of urea fertilizer plants with the potential impact of causing environmental pollution is the disposal activities of wastewater into the waters. The efforts to improve wastewater treatment by separating ammonia have been done by using variety of methods such as: *Ion Exchange* [20], *breakpoint chlorination* [12], *aerobic-nitrification and anaerobic-denitrification electron* [1], *water striping* [29], *fluidized-bed reactor* [15], *anammox (Anaerobic Ammonium Oxidation)* [21]-[23], *combined nitrification-denitrification and microalgae* [25], *membrane reactors (MBR)* [28]. These ammonia separation methods have limitations, such as not being able to reduce the amount of ammonia up to safe concentration level, needing huge cost and the application of some of these methods in practice still encounters obstacles. This constraint is mainly due to the specific capacity of $\text{NH}_3\text{-N}$ removal is still so low that the output process is still higher than the quality standards that have been set.

According to [10] biological waste treatment (microbes) will not run optimally or will be impaired if toxic chemical compounds are found in the wastewater that affect the performance of a waste treatment facility. In the treatment of the wastewater of the urea fertilizer plant, the treatment is carried out by combining chemical and biological processes. The chemical processing technology applied in this study is Advanced Oxidation Process (AOP) or advanced oxidation processes of wastewater treatment which is a chemical processing technology with advanced oxidation using strong oxidator. This process of oxidation can be followed by biological processes involving microorganisms such as microalgae *Chlorella pyrenoidosa*, *Spirillum volutans* sp. and the bacterium *Pseudomonas*.

II. MATERIALS AND METHODS

Material and Tools

Materials used in this study are volumetric flask, pH meter, pipette, Spectrophotometer, scales, aerator, plastic tubing measures ¼ diameters, microscope, fluorescent lamp and the culture media. Petri dishes, transparent millimeter paper. The ingredients needed are water, distilled water, Nessler reagent derived from Wetland area of nitrogen *C. pyrenoidosa* and *Nannochloropsis* sp. pure cultures in the uncontaminated condition or other organisms. Water used is a mixture of sea water and fresh water) as the main culture of *Nannochloropsis* sp with 3% salinity, while *C. pyrenoidosa* using fresh water, pH 8 - 9.5, temperature of 25-30 ° C. Bacteria *P. fluorescens* seeds in the uncontaminated condition. The composition are protease peptone 10 g, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ 0, 75 g, glycerol 7, 5 ml, drilled measurement including pH, density, nitrogen, H_2O_2 , $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, Measurement of $\text{NH}_3\text{-N}$, urea, nitrate, nitrite, TKN, COD,

Research

from a pool emergency tube inserted into the wastewater is analyzed for nitrate, nitrite, TKN, pH, COD, TSS) inserted control samples (waste water) into 5.000 (reagent tubes). The tube serves as a control to react with the fertilizer plant wastewater and various comparisons are $\text{FeSO}_4 : \text{H}_2\text{O}_2$ 1: 1, 1: 2, 1: 3, 1: 4, 1: 5, 1: 10. The samples are stirred at 100 rpm stirring speed setting for 10 minutes, and then the samples were taken after 10 minutes. Then the waste water from the feed tube in the reagent ($\text{NH}_3\text{-N}$, urea, nitrate, nitrite,, TKN, COD, TSS).

- 1 Waste water from in vitro reagent, flowed into aquarium / aquaria (aquarium microalgae, bacteria, bacteria + microalgae), the aerobic process. Later on leave for 7-9 days, because the growth of microorganisms reach stationary phase at 4-6 days. So that microorganisms (bacteria and microalgae) can decompose organic substances contained in waste water
- 1 Water processed, the water that comes from aquarium / aquaria, then analyzed ($\text{NH}_3\text{-N}$, urea, nitrate, nitrite, TKN, pH, COD, TSS), which is useful to know the quality of the waste water from some of the previous process .

C. Circuit Research Tool

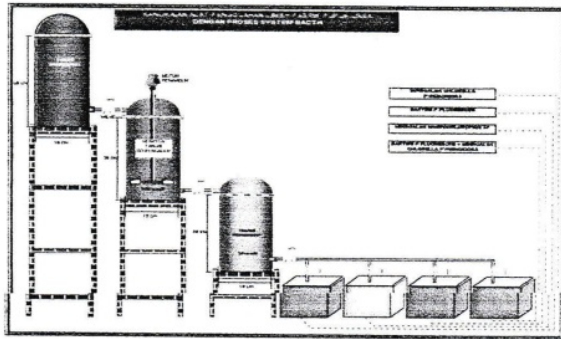


Fig 1. Circuit Research Tools

III. RESULTS AND DISCUSSION

Fenton Reagent Result

The treatment of the wastewater of urea fertilizer plant using Fenton reagent of various concentrations and comparisons of FeSO_4 and H_2O_2 causes a decline in $\text{NH}_3\text{-N}$, urea, COD, TKN, and TSS, but an increase in levels of pH, nitrate and nitrite as presented in Table 1 . The results of the study show a precipitate $\text{Fe}(\text{OH})_3$ which is reddish brown, because the wastewater of the urea fertilizer plant used is that with the acidity (pH) levels of more than 6 [9]. The decline in the value of $\text{NH}_3\text{-N}$ and urea in the results of the study presented in Table 1, is assumed to decompose to form ions and gases, such as nitrate and nitrite molecule, or nitrogen monoxide. This is in accordance with the findings of [27], that the levels of nitrate and nitrite increase. The levels of COD, TSS decrease, indicating that Fenton oxidation can degrade the value of COD and TSS. According to [2], Fenton reagent as one of the advanced oxidation processes (Advanced Oxidation Process / AOPs) is expected to destroy organic and inorganic pollutants, eliminate color and COD.

TABLE I
RESULT ANALYSIS TREATMENT WASTEWATER UREA FERTILIZER
FENTON REAGENT CONCENTRATION 2500, 2000 AND
1500 PPM

Parameter	Initial Ratio Analysis FeSO ₄ (gram) : H ₂ O ₂ (ml)				
	1 : 2	1 : 4	1 : 6	1 : 8	1 : 10
pH	8.4	8.6	9.1	9.1	9.7
NH ₄ -N	135	35	103.75	124	179.11
Urea	1914	1893	1685	2075	1816
Nitrate	1.2	1.87	9.37	44.75	99.69
Nitrite	0.3	0.076	0.316	2.516	6.08
TKN	1038.3	963.07	892.08	968.095	983.243
COD	130	105	119	138	110
TSS	31	31	31	37	37
Concentration (2000 ppm)	1 : 2	1 : 4	1 : 6	1 : 8	1 : 10
pH	8.2	8.5	8.7	8.7	9.3
NH ₄ -N	0.25	0.15	0.013	0.013	0.0
Urea	693	713	702	702	529
Nitrate	0.010	0.21	6.45	6.35	76.8
Nitrite	0.056	0.039	0.056	0.056	3.89
TKN	333.65	239.55	327.513	327.513	181.533
COD	140	115	121	121	120
TSS	35	35	35	35	39
Concentration (1500 ppm)	1 : 2	1 : 4	1 : 6	1 : 8	1 : 10
pH	8.0	8.2	8.3	8.5	9.0
NH ₄ -N	0.044	0.15	0.091	0.0	0.0
Urea	157.05	713	97	32.45	99
Nitrate	0.034	0.21	3.9	16.25	39.89
Nitrite	0.037	0.039	0.037	0.245	2.7
TKN	73.421	239.55	17.266	15.143	9.0
COD	151	115	150	170	134
TSS	32	35	32	35	30

and Microalgae *Chlorella pyrenoidosa* and *Nannochloropsis* sp.

on the treatment of wastewater of the urea fertilizer using Fenton reagent and microalgae *Chlorella pyrenoidosa* and *Nannochloropsis* is a further research. The results Fenton reagent which indicates the level of increasing levels of nitrate and nitrite at a concentration of 2500 ppm, 2000 ppm and 1500 ppm. This level of NH₄-N and urea is expected to be absorbed by microalgae *Chlorella pyrenoidosa* and *Nannochloropsis* sp. nutrients. The results of the study in Tables 2 and 3 show no changes in pH value. The results of the study using microalgae *Chlorella pyrenoidosa* and *Nannochloropsis* sp, no increase or decrease in the pH value is relatively stable during the treatment. The pH did not increase due to the presence of a buffer system in the form of dissolved CO₂ in the culture medium. The dissolved CO₂ in the medium will become carbonic acid which dissociates into ions. The results of the study presented in Tables 2 and 3 show the increase of COD and TSS values. This is due to an accumulation of organic materials in the wastewater resulting from the growth of microalgae *Chlorella pyrenoidosa* and *Nannochloropsis* sp.

TABLE II
RESULT ANALYSIS TREATMENT WASTEWATER UREA FERTILIZER PLANTS
FENTON REAGENT AND MICROALGAE *CHLORELLA PYRENOIDOSA*
CONCENTRATION 2500, 2000 AND 1500 PPM

Parameter	Initial Ratio Analysis FeSO ₄ (gram) : H ₂ O ₂ (ml)				
	1 : 2	1 : 4	1 : 6	1 : 8	1 : 10
pH	8.4	8.6	9.1	9.1	9.7
NH ₄ -N	135	35	103.75	124	179.11
Urea	1914	1893	1685	2075	1816
Nitrate	1.2	1.87	9.37	44.75	99.69
Nitrite	0.3	0.076	0.316	2.516	6.08
TKN	1038.3	963.07	892.08	968.095	983.243
COD	130	105	119	138	110
TSS	31	31	31	37	37
Concentration (2000 ppm)	1 : 2	1 : 4	1 : 6	1 : 8	1 : 10
pH	8.2	8.5	8.7	8.7	9.3
NH ₄ -N	0.25	0.15	0.013	0.013	0.0
Urea	693	713	702	702	529
Nitrate	0.010	0.21	6.45	6.35	76.8
Nitrite	0.056	0.039	0.056	0.056	3.89
TKN	333.65	239.55	327.513	327.513	181.533
COD	140	115	121	121	120
TSS	35	35	35	35	39
Concentration (1500 ppm)	1 : 2	1 : 4	1 : 6	1 : 8	1 : 10
pH	8.0	8.2	8.3	8.5	9.0
NH ₄ -N	0.044	0.15	0.091	0.0	0.0
Urea	157.05	713	97	32.45	99
Nitrate	0.034	0.21	3.9	16.25	39.89
Nitrite	0.037	0.039	0.037	0.245	2.7
TKN	73.421	239.55	17.266	15.143	9.0
COD	151	115	150	170	134
TSS	32	35	32	35	30

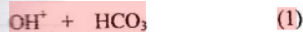
High COD level indicates an organic waste pollution. The inorganic materials found are in the form of clay and sand, and organic matters are in the form of the remains of plants and other biological solids such as algae cells, bacteria and so forth. Based on the results of the study, it can be concluded that the rise in TSS levels is due to the growth of microalgae *Chlorella pyrenoidosa* and *Nannochloropsis* sp.

TABLE III
RESULT ANALYSIS TREATMENT WASTEWATER UREA FERTILIZER PLANTS FENTON REAGENT AND MICROALGAE *NANNCHLOROPSIS* SP.
2500, 2000 AND 1500 PPM

Parameter	Initial Ratio Analysis FeSO ₄ (gram) : H ₂ O ₂ (ml)				
	1 : 2	1 : 4	1 : 6	1 : 8	1 : 10
pH	8.4	8.6	8.9	9.1	9.7
NH ₄ -N	162.25	98	119.2	174	131
Urea	2195	1807	1884	2132	1895
Nitrate	2.53	3.50	13.35	47.75	109.69
Nitrite	0.921	0.99	1.967	3.315	6.31
TKN	1021.95	933.2	775	912.00	1041.36
COD	178	170	166	231	159
TSS	50	50	37	50	53
Concentration (2000 ppm)	1 : 2	1 : 4	1 : 6	1 : 8	1 : 10
pH	8.0	8.2	8.3	8.5	9.0
NH ₄ -N	0.052	0.0	0.002	0.0	0.0
Urea	326	296	138	61.04	1.56
Nitrate	1.94	1.34	6.35	19.93	31.15
Nitrite	0.470	0.774	0.567	1.10	1.67
TKN	156.02	103.96	61.302	29.535	0.725
COD	205	263	167	197	177
TSS	52	52	52	52	52

Pseudomonas fluorescens

water using bacteria *Pseudomonas* research, in which waste water of urea fertilizer plants was treated using Fenton reagent of various concentrations from 2500 ppm, 2000 ppm and 1500 ppm. It still contains high levels of nitrate and nitrite. The levels of nitrate and nitrite presented on Table 4 show changes in pH, namely a decline. The reason is that out by the bacteria that produce CO_2 in the water will shift the carbonate equilibrium that it will lower the pH value. The reaction of the carbonate:



of CO_2 , the reaction equilibrium will shift from the H^+ ions that will cause a decrease of pH. This is in accordance with the opinion of [16], an increase in CO_2 will lower the pH value of the water. In the increase in maintenance time, the amount of CO_2 in the media is also allegedly increased. The CO_2 in the media is also allegedly used as a source of energy for the bacteria. In correlation with the nitrogen to carbon ratio, the addition of carbonic materials, the bacteria will use organic matter and respiration of inorganic nitrogen (ammonia) which is toxic

TABLE IV.
EFFECT OF FENTON REAGENT AND PSEUDOMONAS FLUORESCENS ON THE pH OF WASTEWATER UREA FERTILIZER PLANTS WITH DIFFERENT CONCENTRATION 2500, 2000 AND 1500 PPM

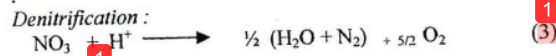
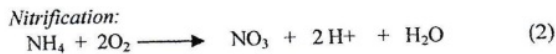
Initial Ratio Analysis	FeSO ₄ (gram)	H ₂ O ₂ (ml)		
1:2	1:4	1:6	1:8	1:10
8.3	8.4	8.5	8.9	9.7
76.5	55	7.5	107.25	119.56
1080	1080	1495	1479	1770.69
0.20	0.98	0.67	32.78	92.45
0.088	0.076	0.043	1.087	6.98
894.18	727.35	697.91	727.44	928.60
13	11	18	17	15
10	12	11	13	10
1:2	1:4	1:6	1:8	1:10
7.8	8.2	8.3	8.7	9.2
0.125	0.08	0.007	0.0	0.0
305	306	693.67	583.07	300.4
0.04	0.34	0.56	22.08	69.90
0.074	0.034	0.023	1.024	3.053
270.90	232.84	323.782	272.09	12.290
15	15	15	19	14
14	15	15	16	14
1:2	1:4	1:6	1:8	1:10
7.7	7.8	8.0	8.3	8.6
0.075	0.003	0.0	0.0	0.0
248	15	145.78	1.056	0.0
0.02	0.26	0.91	17.45	69.90
0.064	0.019	0.011	0.019	0.12
48.95	6.093	0.207	0.482	0.0
20	15	23	19	25
15	17	15	17	15

The addition of carbonaceous material is proven to be effective in reducing inorganic nitrogen [2]. Bacterial growth is affected by the balance of nutrients in the water. Therefore, the

dynamics of bacteria population are closely related with the availability of nutrients [10]. The data in Table 4, show that the levels of nitrate and nitrite decrease. The decrease is due to the fact that the nitrate and nitrite formed are sufficient for bacterial nutrients that will stimulate the growth of bacteria and the increase of bacterial biomass. Although in low concentrations, nitrite is toxic to fish and other aquatic organisms [13]. Nitrite compounds in fish will be bound in blood that will form methaemoglobin ($\text{Hb} + \text{NO}_2 = \text{Met-Hb}$). The Met-Hb would interfere with the transport of oxygen to the tissues of fish that can cause fish to experience hypoxia. The Met-Hb in the blood will cause the blood to become brown. Therefore, nitrite poisoning is also called brown blood disease [4][1][24]. The levels of Total Suspended Solid (TSS) and Chemical Oxygen Demand (COD) increase which indicates that there is accumulation of organic materials in the wastewater derived from *Pseudomonas fluorescens* bacteria growing in the waste water of urea fertilizer plants, which is in a state of non-toxic. Whereas the increase of TSS level is due to the occurrence of wastewater suspended solids which form residual components, floating materials, and suspended colloidal components. Suspended solids contain inorganic and organic materials. The inorganic materials are in the form of clay and sand, while the organic matters are in the form of the remains of plants and other biological solids such as algae cells, bacteria, and so forth [17].

D. Fenton Reagent, Microalgae *Chlorella pyrenoidosa* and Bacteria *Pseudomonas fluorescens*

In further processing of the treatment of the waste water of the urea fertilizer plants which contains high levels of ammonia and urea using Fenton reagent, it is expected that it will be continued with the use of a combination of microalgae *Chlorella pyrenoidosa* and bacteria *Pseudomonas fluorescens*. The microalgae and the bacteria will work together in upgrading the waste water. The results of this study presented on Table 5, in general show that pH values tend to decrease with the increasing of maintenance time. This fluctuating value is allegedly due to the addition of the nitrification and denitrification bacteria into the maintenance media. The mechanism of bacterial nitrification and denitrification which can affect pH level can be described by the following equation [17].



Through this equation it can be seen that nitrifying bacteria in the process to convert NH_4^+ (ammonium) to NO_3^- (nitrate) produce H^+ ions that can make the pH of the maintenance medium drop. Since in a large-scale microalgae always associate with bacteria, the interaction between algae and bacteria will be able to purify river water. Metabolic activities of heterotrophic aerobic bacteria produce CO_2 , NH_4 , NO_3 , PO_4^{3-} , and so on. The microalgae absorb those

and produce organic matters, O₂, and H₂O. The produced by microalgae is used by heterotrophic bacteria for the reaction of nitrification and is used by bacteria for denitrification. Through the process of synthesis, microalgae using CO₂ from aerobic bacteria form cell protoplasm and release oxygen

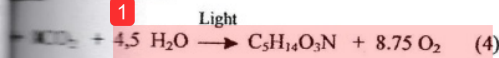


TABLE V.
ANALYSIS TREATMENT WASTEWATER UREA FERTILIZER WITH FENTON REAGENT AND MICROALGAE *CHLORELLA* AND *PSEUDOMONAS FLUORESCENS* BACTERIA 2500, 2000 AND 1500 PPM.

Initial Ratio Analysis FeSO ₄ (gram) : H ₂ O ₂					
1 : 2	1 : 4	1 : 6	1 : 8	1 : 10	
8,2	8,3	8,8	8,8	9,7	
22,34	73,12	96,43	103,89	125,78	
1856	1620	1421	1500,03	1823,23	
0,97	0,57	0,56	41,43	103,89	
0,076	0,767	0,230	1,767	6,076	
853,88	746,13	663,56	763,90	986,62	
101	93	100	115	100	
59	59	59	59	59	
1 : 2	1 : 4	1 : 6	1 : 8	1 : 10	
7,8	8,9	8,3	8,7	9,2	
0,375	0,078	0,094	0,0	0,0	
597	520	485	660,65	0,0	
0,73	0,230	0,61	21,23	32,98	
0,062	0,729	0,096	0,729	0,97	
273,995	239,278	226,33	308,30	0,0	
136	99	105	119	114	
60	60	60	69	62	
1 : 2	1 : 4	1 : 6	1 : 8	1 : 10	
7,7	7,9	8,1	8,3	8,7	
0,025	0,003	0,001	0,0	0,0	
205	59	0,75	0,0	0,0	
0,26	0,86	0,98	16,98	32,98	
0,056	0,597	0,074	1,597	0,97	
94,32	2,309	0,346	0,0	0,0	
138	101	111	125	114	
62	50	62	62	62	

The main source of nitrogen that can be used by microalgae are nitrate and ammonia-N. The bacteria utilize the materials produced by or derived from dead microalgae as a carbon source for the synthesis of new cells and to obtain the energy to form the final product such as CO₂, during the process of respiration and synthesis. Microalgae use CO₂ as a carbon source for photosynthesis.

IV. CONCLUSION

The best ratio between FeSO₄ : H₂O₂ in treating waste water of urea fertilizer plant using traditional Fenton reagent which meets the quality standards of waste water quality issued by the Minister of Environmental Affairs of the Republic of Indonesia No.122 of the year 2004 and the Decree of South Sumatra Governor No.18 of the year 2003 is 1 : 4, with a concentration of 1500 and 2000 ppm. The treatment of waste water of urea fertilizer plants by means of advanced oxidation using microalgae *Chlorella* *sp.*, *Nannochloropsis* *sp.*, *Pseudomonas fluorescens* and the synergy between microalgae *Chlorella* *sp.* and bacteria *Pseudomonas fluorescens* results in a decrease of pH, NH₃-N, Urea, TKN, Nitrate and Nitrite and an increase of levels of TSS and COD. And the best treatment was using *Pseudomonas fluorescens* bacteria.

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