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NH_3-N and COD Reduction in Endek (Balinese Textile) Wastewater by Activated Sludge under Different DO Condition with Ozone Pretreatment

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Abstract

Nitrogen and organic matter are part of the pollutant causing eutrophication in freshwater. Textiles industry like Endek is the main source of Nitrogen and organic matter. Tis paper aims to know the degradation process of Ammonia-Nitrogen (NH_3-N) and Chemical Oxygen Demand (COD) with ozone pretreatment in operated by different DO level activated sludge. There are 2 scenarios of treatments in this study: with ozone pretreatment (R1) and without ozone pretreatment (R2). Wastewater treatment began with the seeding and acclimatization process. This acclimatization study showed the efficiency degradation of NH_3-N and COD by 17.7 and 27.5 %. Biological Oxygen Demand (BOD)/COD level increased with ozone pretreatment, from 0.25 to 0.38, COD/TKN level stated at 3.26. Ozone pretreatment reduced NH_3-N and COD by 23.8 and 34.1 %. Wastewater treatment with activated sludge operated by different DO levels showed efficiency of 44.2 % (R1) and 68.2 % (R2). This ammonia elimination was capable of preventing eutrophication in the waterbody. The efficiency of NH_3-N and Nitrogen organic degradation was indicated by TKN (Total Kjeldahl Nitrogen) levels: 87 % (R1) and 79 % (R2). The concentration of Nitrate (NO_3-N) increased from 2.9 to 5.5 mg/L when DO reached 1 - 3 mg/L. COD reduction levels in this study were 17 % (R1) and 42.5 % (R2). Ozone pretreatment could make the efficiency of wastewater treatment qualified into standard quality.

Keywords: COD removal, Endek wastewater, NH_3-N removal, Ozone pretreatment

Introduction

Industrial growth including the textile industry develops environmental pollution, directly or indirectly [1,2]. Textile wastewater treatment very difficult because of fluctuating pH, colors, temperature, and highly contain total dissolved solids (TDS), total suspended solids (TSS), and total solids (TS) [3]. Textiles wastewaters consist of high organic matter, some metals and complex dyes [4,5]. Azo is organic syntetics compound group and coloured because of azo bonding (-N=N-). Azo is difficult to damage both chemically and photolytic. If azo is disposed to water body, it will disrupt aesthetics and poison the biota in freshwater.

One of the dyes that used azo is endek woven from Bali province, Indonesia. All this time, the endek woven industry has difficulty reducing Nitrogen and organic matter in its wastewater because of azo bonding (-N=N-). The existence of -N=N- waste will block sunlight into the water during the photosynthesis process and cause a reduction of oxygen that will impact the biota's life. Other than that, dye waste that is disposed into the environment will cause eutrophication, dangerous and toxic by-product from a chemical reaction (eg. oxidation, hydrolysis) and decreasing of water quality [6]. Therefore, wastewater treatment for dye waste has an important role not only in environmental pollution but also in the application of technology effectively. Dyes can not be treated by aerobic biodegradation and conventional biological method easily [6].

Textile wastewater effluent only contains 47 % of biodegradable compound [7]. The BOD/COD ratio less than 0.1 shows a very low existence of organic that can be degraded by microorganisms [8]. This ratio can be improved with pretreatment ozone [9]. Wastewater treatment with ozone is difficult to degrade Nitrogen properly. Nitrogen can be degraded with controlling DO (low to high) with an activated sludge reactor.

The decreasing of Nitrate concentration based on the Nitrogen cycle principle, the changing of ammonia into Nitrate to Nitrite by ammonia-oxidation bacterias (AOB) [10], and formation of Nitrogen gas using Nitrite as electron acceptor terminal with the assist of ammonia heterotrophic bacterias (AHB). Nitrite oxidizing bacteria (NOB) are also used to know the oxydation in the reactor. The decreasing of Nitrate concentration is influenced by those bacteria, so that in this study, the efficiency of NH_3, Nitrite or Nitrate, and COD are conducted.

Nitrification partial with Annamox needs 40 % of oxygen from the nitrification process with Carbon absent and low sludge production [11]. This system is well used in wastewater treatment with low organic biodegradable matters and high concentration of ammonium. Anammox feeding reactor needs a ratio of Ammonium to Nitrit 1:1 molar. Nitrification partial can be reached if NOB can inhibit free ammonia when its concentration around 10 - 15 mg/L [12] and DO at low concentration can support the growth of AOB [13]. Some studies show low DO (< 2 mg/L) can reach nitrification partial effectively [14]. The high concentration of COD with organic biodegradable matters more than 500 mg/L support the growth of AHB which is growing faster than autotroph. It causes nitrification autotrophic bacteria easy to overgrow by heterotroph and makes the efficiency of nitrification decreases. The competition between nitrification and heterotrophic bacteria can be improved with high DO.

Tis study aims to know the process of endek wastewater treatment with ozone pretreatment. The main treatment is conducted by using activated sludge that is operated from low DO to high DO so the optimum process to degrade ammonia as Nitrogen (NH_3-N) would be known.

Materials and methods

Experiment set-up

This study used endek wastewater taken from Semarapura, Bali. Endek wastewater was collected in a tank before treated in an ozone and activated sludge reactor. The treatment used a sequencing batch, that consisted of several phases. **Figure 1** shows the experimental system of ozone pretreatment and activated sludge reactor with different DO conditions. The raw endek wastewater in the feed tank was used as the influent with ozone pretreatment (R1) and without ozone pretreatment (R2). The working volume of ozone and activated sludge reactor was 12 L. Ozone reactor connected to an ozone generator. Whereas, activated sludge reactor had dissolved oxygen (DO) meters and diffusers connected to an aerator.

Procedure of analysis

Organic pollutant in textile wastewater treatment represented by parameters. In this study, some of the parameters like VSS (volatile suspended solid), BOD_5, COD (Chemical Oxygen Demand), NH_3-N (ammonia-N), Total Kjeldahl Nitrogen (TKN), NO_2^- (nitrite), and NO_3^- (nitrate) measured by Indonesian National Standard. VSS measured with Gravimetric method, dried at 103 - 105 °C and then transferred to a 600 °C furnace [15]. The BOD_5 (Biological Oxygen Demand) was measured with Standard Method 5210. Follow the procedure outlined in standard method 5220; Ammonia-Nessler method 8038 - Hach; EPA method 1687; standard method 4500-NO_2-B [15]; and standard method 4500-NO_3 [15] for the analysis of samples for COD, NH_3-N, TKN, NO_2^- and NO_3^-.

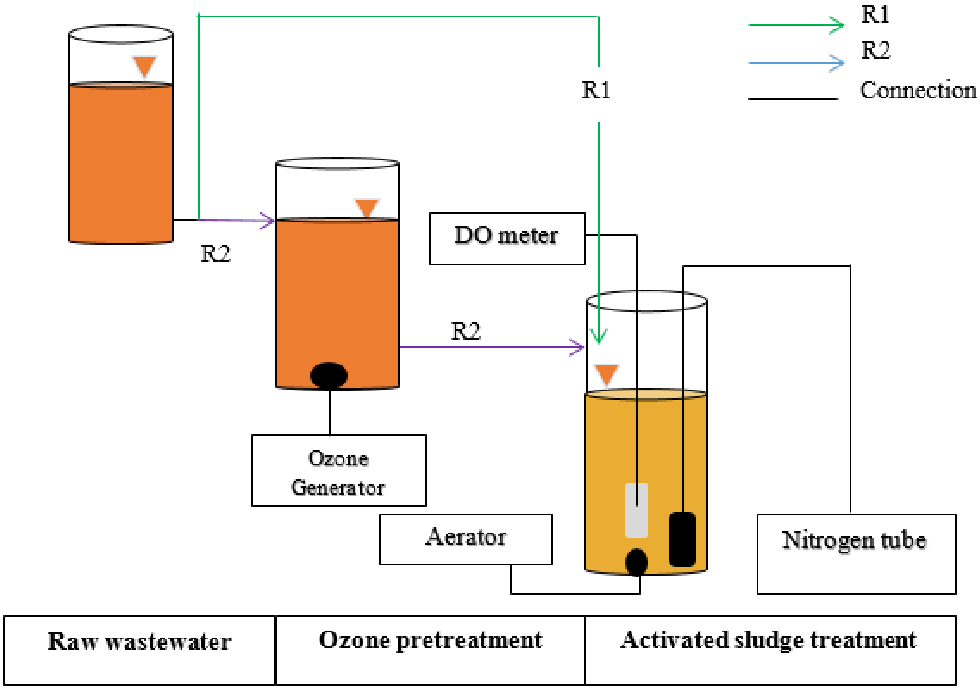


Figure 1 Schematic diagram from raw wastewater and treated to ozone pre-treatment and activated sludge reactor with aerated with different DO condition.

Wastewater treatment

Seeding was conducted anoxically not only with the assist of a diffuser but also the addition of Nitrogen gas. DO level fluctuated daily and periodically depends on the mixing and movement of mass water, photosynthetic activity, microorganism's respiration, and organic matters in wastewater. DO level need to be controlled every day with a nitrogen tube and DO meter (**Figure 1**), about 0.5 - 4 mg/L.

The acclimatization process was done after the seeding process. The acclimatization process aims to increase the number of microorganisms that can be used in the next process, so that microorganisms used endek wastewater as their substrate. There are 4 phases of acclimatization process 20, 40, 60, and 80 % of endek wastewater with the addition of glucose as substrate. These phases aim to give the exact concentration of limiting nutrients [16] in endek wastewater. Phase 20 and 40 % used glucose as the main substrate and tofu waste as the additional substrate. Another phase, involving 60 and 80 % of endek wastewater, was already used as the main substrate.

Ozone pretreatment was conducted by an ozone generator with 0.05 mg/min dose and 60 min of detention time (R1). After treatment in each of the reactors, R1 and R2, the treatment was continued with activated sludge treatment. The runing process in this treatment was done with different levels of DO (low concentration to high concentration).

The initial concentrations of NH3-N, nitrites, and nitrates when treating 100 % of endek wastewater for each reactor were 16.2, 7.9 and 0.6 mg/L for R1 and 12.2, 6.8 and 0.5 mg/L. The initial concentrations for the COD parameters for R1 and R2 were 394.6 and 145.4 mg/L respectively and the initial C/N ratio for each reactor R1 and R2 are 3.21 and 3.26, respectively. Organic biodegradability based on BOD/COD parameters showed less degradable values for R1 (0.25) and degradability for R2 (0.38). At this stage, wastewater treatment is carried out with a detention time of 850 min.

Results and discussion

Acclimatization

The reactor was operated at room temperature (29 ºC) during start-up conditions. Inoculation and acclimatization phase aimed to adjust the microorganism life condition. Microorganisms were added to the reactor and they had to survive with an excess substrate, which is related to NH_3-N and COD level in wastewater. Ratio level of C/N had to be controlled up to 4 in acclimatization phase (**Figure 2**) to ensure the denitrification process was not limited by carbon source [17].

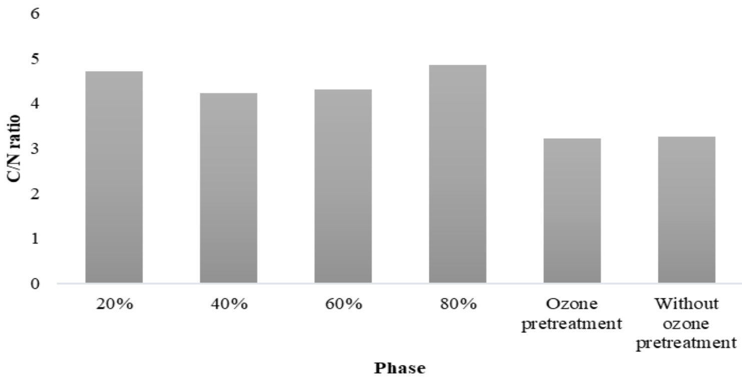


Figure 2 Ratio level of C/N in raw wastewater treatment from acclimatization until activated sludge treatment.

The efficiency of COD degradation was increased gradually with a small range in acclimatization process, 27.5 % (average value). The degradation of NH_3-N showed an average value of 17.7 %. The concentration of NH_3-N decreased due to the degradation process of NH_3-N into NO_2^- and NO_3^-. Besides, nitrification process can be degraded NH_3-N into NO, N_2O, or N_2 gas. The efficiency of COD and NH_3-N degradation during acclimatization process was conducted gradually (**Figure 3**).

Mixed-Liquor Volatile Suspended Solid (MLVSS) is the total amount of volatile solid suspended in the form of organic matters and mineral including microorganism such as organic matters of living or dead microbes and also cell destruction [18,19] The measurement of MLVSS was conducted to ensure reactor operated according to design criteria [20]. During the acclimatization process, MLVSS level increased due to the growth and breeding of microorganisms in the reactor. Increased of MLVSS in this biological wastewater treatment indicated that microorganism can do degradation process of organic matters and Nitrogen [14]. This is supported by the result of acclimatization process which is showed the increasing number of microorganisms. Nevertheless, during the acclimatization process, the rate of growth of microorganisms specifically decreased than the seeding process (**Table 1**).

Table 1 Specific growth rate (μ) from seeding and acclimatization process.

Phase	Specific growth rate (μ) $\left(\frac{1}{day}\right)$
Seeding	0.0091
20 %	0.0087
40 %	0.0044
60 %	0.0032
80 %	0.0045

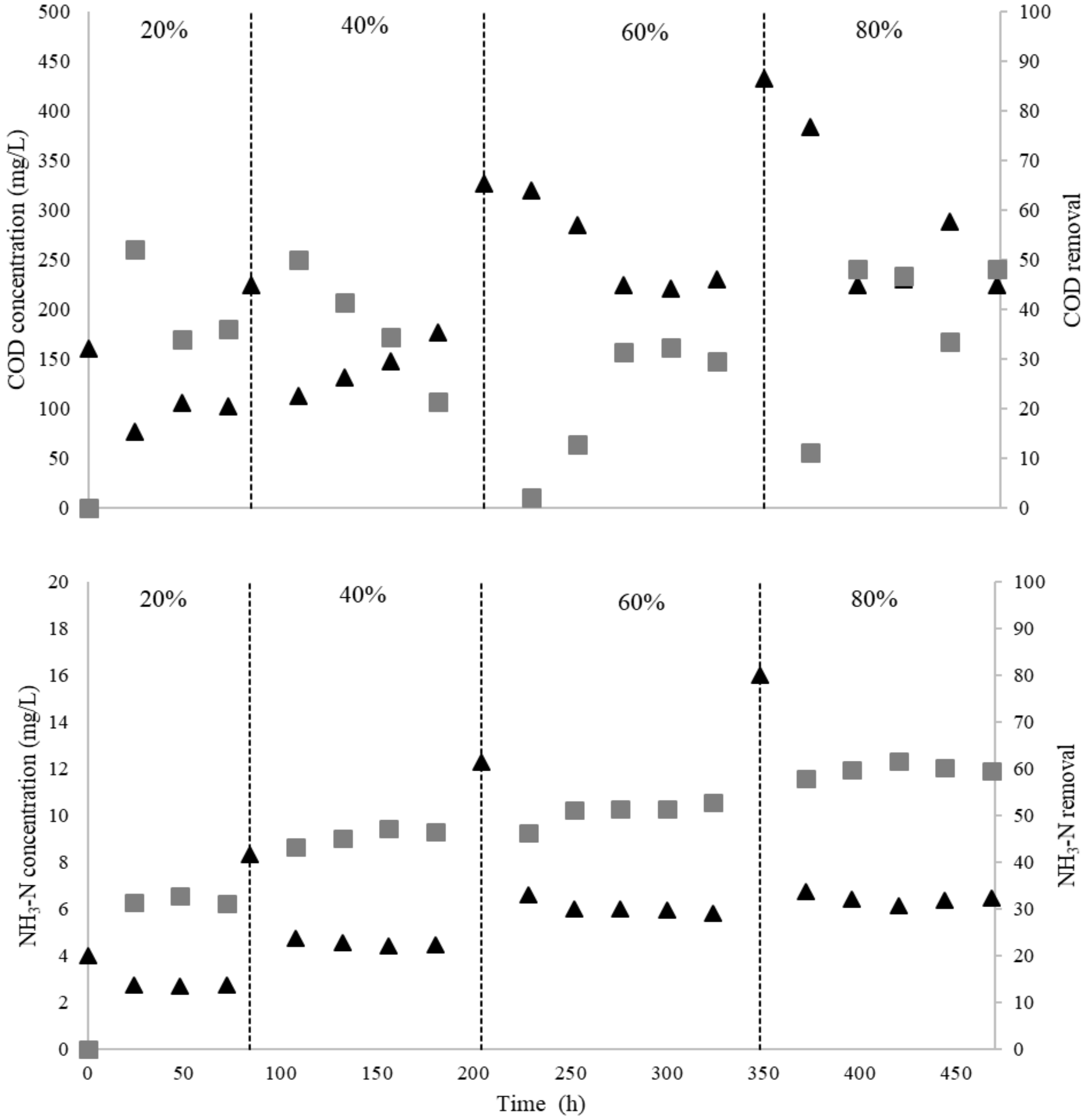
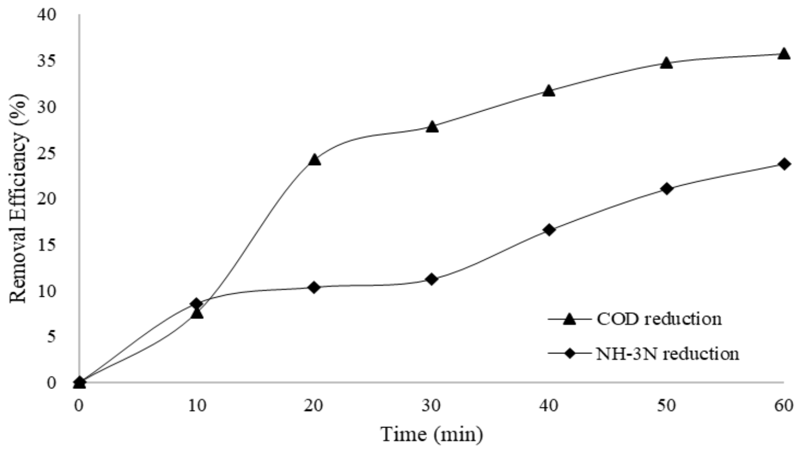


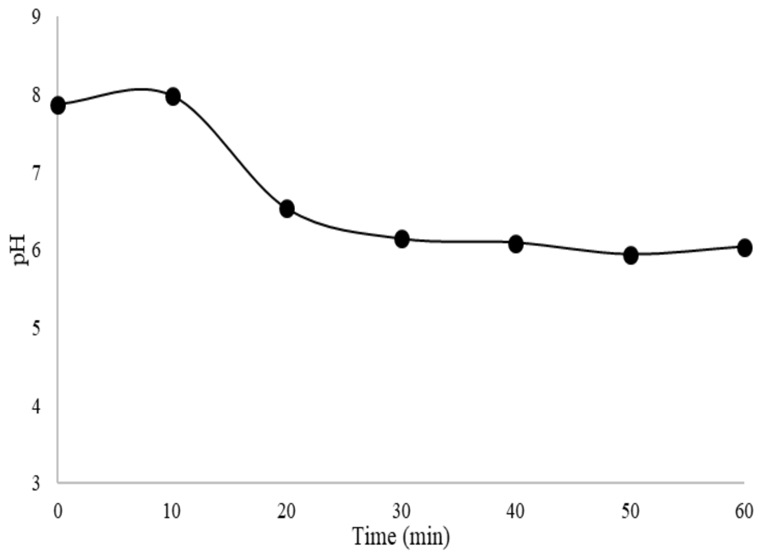
Figure 3 Degradation of COD and NH_3-N in acclimatization process in different concentration of endek wastewater from 20, 40, 60, and 80 % ■ (Concentration) ▲ (Removal efficiency).

Ozonation pre-treatment

The reduction of NH_3-N and COD obtained after ozone treatment was 36 and 24 % (**Figure 4a**). Ozone pretreatment is efficient in COD and NH_3-N removal and can improve with biological wastewater treatment processes. Study of Liang *et al.*, ozone base textile wastewater treatment can remove 20 % of COD and increased the biodegradability to 0.33 - 0.68 [21]. This COD reduction was similar to Suryawan et al [5], the BOD/COD ratio was increased from 0.25 to 0.38. In addition, there was no significant change differences C/N ratio between R1 (3.21) and R2 (3.26). The decrease in COD concentration was contributed simultaneously by ozonation and biological treatment mechanism [22]. Suryawan *et al.'s* result the increase of biodegradability can improve the wastewater effluent quality [20]. The decrease in the pH from 7.85 to 5.43 was observed at the end of the treatment (**Figure 4b**). Decomposition of COD at 20 min of pH was constants at acid value were measured.



(a) COD and $NH_{3\text{-}}$N reduction during ozone pretreatment



(b) pH change during ozone pretreatment

Figure 4 The efficiency of COD and NH_3-N reduction (a) and the change of pH (b) during ozone pretreatment (▲(COD) ♦ (NH_3-N)) ● (pH).

Degradation of NH_3-N

The degradation of NH_3-N at reactor R1 and R2 gave efficiency by 44.2 and 68.2 %. The use of sugarcane activated carbon (SCAC) could degrade NH3-N by 41.05 % [17], this value shows similarity with the R1 reactor's result. Micro-peat (M-P) as absorbent degraded NH_3-N by 65 % [23], and similar with R2 reactor's result. The efficiency of nitrogen removal in leachate in the study Miao *et al.*, through an aerobic-anoxic process it reaches 80 % with initial concentration NH_3-N and NO_3 each of 2000 mg/L and 0.5 mg/L [24]. With this ammonia allowance, it can prevent harm for fishes and stimulate algal growth [25].

The concentration of Nitrate in this study increased when the level of DO increased (**Figure 5**). Nitrate was the main formation of Nitrogen in natural waters and nutrient for plants and algae. It occurred because microorganisms did not utilize nitrate that is formed from the decomposition of organic matters (Nitrogen), so Nitrate became accumulated in the reactor. Dissolved oxygen concentration and a lower concentration of nitrate-nitrogen in water of lake can decrease input of organic matter [26].

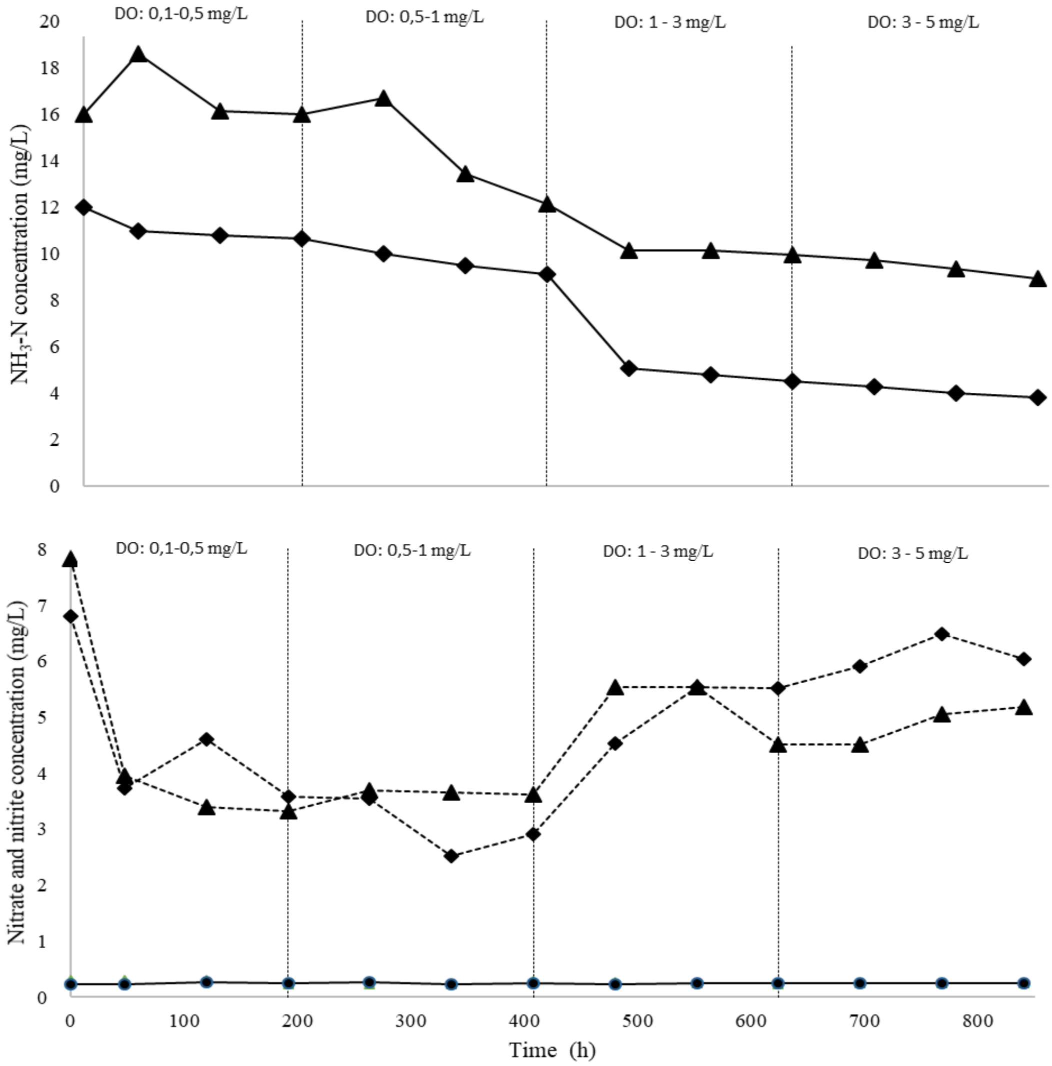


Figure 5 Degradation of NH_3-N, Nitrite, and Nitrate in different DO level from activated sludge reactor ▲(R1) ♦ (R2).

Whereas TKN as a total concentration of organic nitrogen and ammonia level, showed removal of 87 % (R1) and 79 % (R2). TKN was readily degraded under the different DO concentration conditions from low to high concentration during react, which are 16.6 mg/L (R1) and 39.5 mg/L (R2). This TKN level is lower than Sirianuntapiboon and Yommee's study, 4.1 mg/L [27]. **Figure 5** showed the degradation of NH_3-N optimum at the DO level > 3 mg/L but decreased when the DO level < 3 mg/L. Ammonia-oxidation bacteria (AOB) were dead when DO reached a high level, which is above 3 mg/L. However, that high level of DO caused ammonia heterotrophic bacterias (AHB) rate growth increase and support the degradation of COD properly [28]. In fact, when NH_3-N degradation had reached the optimum level, the concentration of NH_3-N decreased statically; in other words, it was saturated.

The concentration of NH_3-N had fluctuated due to the decomposition of organic matters in endek wastewater (Nitrogen formed) into NH_3-N. High-level DO made ammonia transformed into Nitrate and Nitrite. However, low level DO increased NH_3-N concentration and it was also caused by ammonia accumulation as the result of organic matter decomposition by bacteria. Besides, the concentration of ammonia also increased due to the decomposition of old and dead microorganisms. Old microorganisms existed because of activated sludge's age (Θ_c) over the limit (more than a week since acclimatization process) and incapability to regeneration new microorganism (related to wastewater quality as growing media). When Θ_c was more than 30 days, the efficiency disclosed no further improvement [29]. It showed a low rate growth of microorganisms in **Table 1**.

COD degradation

Degradation and reduction of organic matters in endek wastewater showed with the decrease in COD concentration. COD decreased effectively increased when the DO level was increased (**Figure 6**). The pretreatment ozone can reach the COD removal up to 42.5 % with initial and effluent COD concentration of 394.6 mg/L and 145.4 mg/L. The COD result is in good agreement with the findings of previous studies [20]. As one can see from these data, the organic dynamics for dyes are practically the same. This depends on the chemical structure of organics and the stoichiometry of the reaction dyes with ozone and microbes. In sewage treatment with filtration process can remove COD from 48 to 64 % depending on hydraulic load [30].

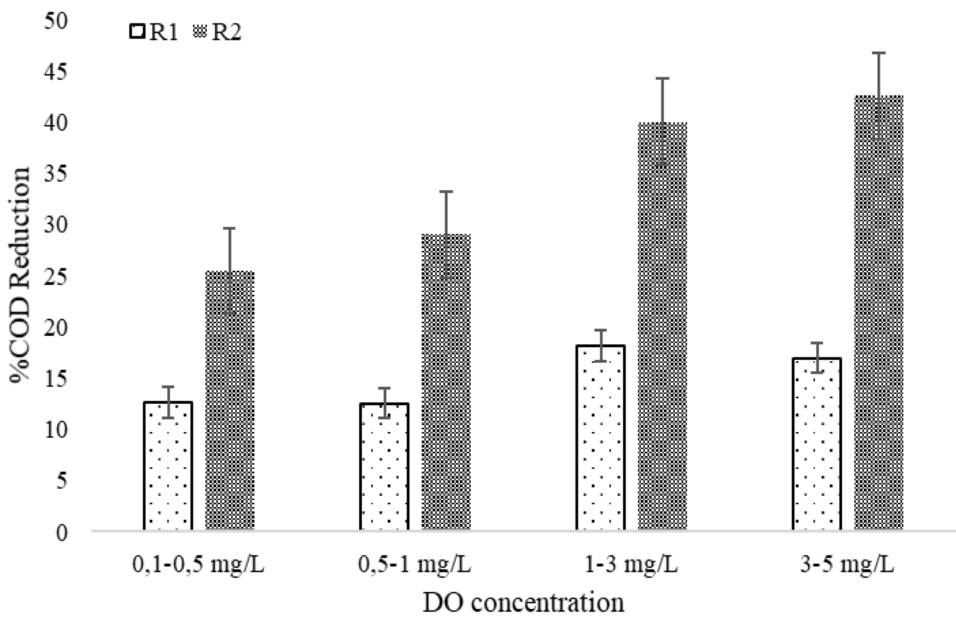


Figure 6 Reduction efficiency of COD in different DO levels from activated sludge reactor.

The result Jóźwiakowski, the COD substance removal in a hybrid sand filter for domestic wastewater is 72.3 % [31]. This result is higher due to the higher load in dye wastewater treatment. Decomposition of organic matters by microbes will produce inorganic matters as nutrients that is also used by microbes. It showed from the changing in nutrients level.

Advantages and disadvantages

The application of wastewater treatment at this time needs to pay attention to the quality of effluents produced in the nutritional parameters. Roman *et al.*, stated that the fluctuating of Nitrogen (N) and Phosphor (P) nutrient is caused not only by dead algae but also algae growth, N, and P concentration in water and concentration of N and P that is contained in algae [32]. Nutrient concentration in textile wastewater with conventional treatment shows unsatisfactory value, especially on the NH_3-N parameter and the dense color in wastewater [33,34].

Treatment with ozone technology can remove colors very well and the water can be reused [33,35]. Colored water will cause community discomfort because the aesthetics of the color in the body of water turn concentrated. A positive aspect of the application of this technology is the increase in effluent quality in wastewater. Water treatment based on pilot-scale ozone-biofiltration without reverse osmosis has been carried out and gives positive results, where microorganisms are not detected [36]. Wastewater treatment with ozone as a pre-treatment integrated with biological activated carbon (BAC) is lower cost and operation and maintenance (O&M) compared to reverse osmosis (RO) [37].

Further research for wastewater treatment with the ozone pre-treatment process needs to be done in wastewater treatment. Pilot-scale has been applied to textile waste treatment with 90 % of color removal [38]. However, it is necessary to conduct research for pilot scale to treating endek wastewater especially ozone as pre-treatment with activated sludge. The use of ozone technology also requires energy to supply gas into the reactor continuously. This condition causes an increase in operational costs. On the other hand, wastewater treatment demands a low cost and high treatment efficiency [20].

Conclusions

Biodegradability in ozone pretreatment can increase from 0.25 to 0.38. Ozone pretreatment is suitable for advanced treatment of achieving NH_3-N and COD removal efficiency higher than without ozone pretreatment. The effluent NH_3-N are 8.9 and 3.8 mg/L and the COD reduction is 17 and 42.5 %. The simultaneous DO concentration could be achieved in the activated sludge treatment, which enhances NH_3-N and COD removal. This application is highly recommended in developing countries that still use biological treatment to treat wastewater that has low biodegradability. The application of this technology must be tested by increasing the scale in laboratory-scaleup research before it is applied to the field scale.

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