Treatment of Tofu Industry’s Wastewater Using Combination of Ozonation and Hydrodynamic Cavitations Method with Venturi Injector

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Abstract. Indonesia is one of the country with the biggest population of tofu industries in the world. Tofu wastewater can harm the environment and alter the physical and chemical properties of water in river bodies, which is dangerous for human and living organism within the river. So it needs to be treated before disposed into the environment. The purpose of this research is to evaluate the performance of ozonation [O], hydrodynamic cavitations [HC], and combination of both [HC/O] in treating tofu wastewater. The variations of circulation flow rate are 2, 3, and 4 LPM and dosage of ozone is single and series ozonator. [HC/O] method (4 LPM flowrate and 129 mg/h dosage of ozone) produces the best performance, with degradation of 943 mg/L COD and 293 mg/L TSS in 180 minutes. This result is much better than the 4 LPM variant of [HC] method and series variant of [O] method separately, which are only capable of degrading 485 mg/L COD-288 mg/L TSS and 136 mg/L COD-233 mg/L TSS in 180 minutes accordingly.

Keywords: COD, hydrodynamic, ozonation, tofu wastewater, TSS

1. Introduction
Indonesia is one of the country with the biggest population of tofu industries in the world. According to the data from Indonesia’s Ministry of Technology and Research in 2010, there are a total of 84,000 tofu industries with a combined production capacity of 2.56 million tons per year, and 80% of that number is located in Java Island. From the same source of data, it is known that these industries produce as much as 20 million m³ wastewater annually [1].

Tofu wastewater is a pollutant that can harm the environment, it can alter the physical and chemical properties of water in river bodies, which is dangerous for human and living organism within the river, if no treatment process is taken against it. Tofu wastewater is known to have a high content of Biological Oxygen Demand (BOD), Chemical Oxygen Demand (COD), and Total Suspended Solids (TSS) [2]. Tofu wastewater is known to have characteristic of acidic pH (4-5), BOD value of 6,000-8,000 mg/L, COD value of 7,500-14,000 mg/L, and TSS value of 6,000-8,000 mg/L [3]. These high value of BOD, COD, and TSS characterize the harmfulness of tofu wastewater towards the environment. For that reason, the Indonesia’s Government through the Ministry of Environment announced the allowable dumping quality for tofu wastewater which is BOD <150 mg/L, COD <300 mg/L, TSS < 200 mg/L, and pH 6-91 [4].
Efforts have been done in order to treat and lower the pollutant parameters value of tofu wastewater, amongst them are anaerobic treatment and anaerob-aerobic biofiltration. Both of these treatment methods are still known to have a low effectiveness, where each of anaerobic treatment and anaerobic-aerobic biofiltration has to be operated for as long as 4 weeks and 2 months accordingly for the effluent to the allowable dumping quality [5]. Many studies have been done in search for a more effective alternative method with relatively faster process duration. One of the alternative method that gains popularity is Advanced Oxidation Process (AOP) technology based method, or ozonation and hydrodynamic cavitation specifically.

Ozonation and hydrodynamic cavitation are types of AOP technology used in many applications of organic wastewater treatment process. Ozonation is known to have the capability of reducing 40-50% of COD within the organic wastewater [6]. While the hydrodynamic cavitation using vortex diode has capability of reducing 61% of COD within methyl red dye wastewater [7].

By combining hydrodynamic cavitation with ozonation, the oxidation process of organic pollutant within wastewater could be maximize. Combination of both method is known to have the capability to increase the effectiveness of total coliform disinfection from 60 to 80% [8]. This research has purpose to evaluate the performance of ozonation, hydrodynamic cavitation, and combination of both methods in decreasing the value of BOD, COD, and TSS within tofu wastewater, by making variation on the flow rate and dosage of ozone being used.

2. Basic Theory

2.1. Pollutant Parameters of Tofu Wastewater
BOD, COD, and TSS are pollutant parameters, which values are usually used to define the quality of tofu wastewater and decide the possibility of it to be introduced to the environment. BOD is an empirical analysis used to globally predict the microbiology processes take place in water. The value of BOD defines the concentration of oxygen that is needed by the microorganisms to oxidize almost all of the soluble organic and part of suspended organic substances in water. On the other hand, COD is the concentration of oxygen that is needed to oxidize organic substances within a sample/water by using oxidant such as K2Cr2O7 and KMnO4 [9]. High value of BOD and COD indicate a huge pollution load within water, which need to consume a high concentration of dissolved oxygen in water for the oxidation process to occur. BOD and COD within tofu wastewater origin from the leftover of soybean proteins, carbohydrates, and fats from tofu production processes, with protein as the dominant content [10].

TSS is an analysis used to calculate the content of suspended solid particulates within water. In huge amount, suspended solids could increase the turbidity of water, which blocks the penetration of sunlight through water and in turn interfering with the activity of producer organisms within water. TSS within tofu wastewater origin from the soybean solid particulates which are not filtered during the making processes and the naturally occurred microorganisms [10].

2.2. Advanced Oxidation Process (AOP)
Advance Oxidation Process (AOP) is a compilation of chemical oxidative technologies which are popular amongst the researchers and industry practitioners during these last three decades. All of the AOP technologies have a characteristic of OH radicals producing processes, the strongest oxidant in liquid medium that is highly reactive and non-selective [11].

The oxidation is defined as a movement of one or more electrons from a reductant to an oxidant, which in several cases produce chemical species with odd valence of electron named...
Radicals. Radicals have unstable and highly reactive characteristics, which make them good as an advanced oxidation initiator. Specifically, AOP is a process where the oxidation is primarily/dominantly done by OH radicals [12]. OH radicals have the ability to oxidize almost all type of organic compounds into water, carbon dioxide and mineral through a mineralization process [13].

In its application, AOP involves ozone (O₃), hydrogen peroxide (H₂O₂), UV light, TiO₂ catalyst, cavitation, irradiation of E light, and Fenton reaction, which all have capability to produce OH radicals. Ozonation and hydrodynamic cavitations are the types of AOP used in this research.

2.2.1. Ozonation. O₃ is a strong oxidant in which produced naturally through the ionization of air by lightning or artificially through ionization of oxygen (O₂) by high voltage electricity. Nowadays, O₃ is used in many applications of disinfection, oxidation of inorganic compounds, and oxidation of organic compounds for water and wastewater.

O₃ could react with other chemical species through two kinds of reaction chains, the direct and indirect reaction chain. Those two kinds of reaction chains are controlled by different kinetics and also produce different final products. The indirect reaction chain involves radicals, a non-selective and highly reactive oxidant. The indirect reaction chain could be divided into three steps: the initiation, chain propagation, and termination. Initiation is a step where O₃ is broken down into secondary oxidants, e.g. OH radicals. The produced OH radicals will then react with other chemical molecules/substances (target molecules) directly and non-selectively (k = 10⁶-10¹⁰ M⁻¹ s⁻¹) to produce water. From that reaction, the target molecules will lose one of its electrons and transformed into radicals. That radicals will further react with other target molecules, which is known as chain propagation step. The termination step is achieved when the radicals attack other radicals which neutralize and stop the reaction [14].

In direct reaction chain, the organic compounds are oxidized directly by O₃. This direct reaction chain is selective and relatively slower than the indirect one (k = 1,0-10⁶ M⁻¹ s⁻¹). O₃ molecules react with the non-saturated bond through a Criegee mechanism, as shown in Figure 1 [14].

2.2.2. Hydrodynamic Cavitation. Hydrodynamic cavitations occur and spread inside a water jet flowing through a channel whose geometry causes a variations of static pressure in the flow [15]. The mechanism of hydrodynamic cavitations within venturi is illustrated in Figure 2. When a liquid (preferably water) flows through the inlet of venturi, the cavitation nuclei (in the form of 10⁻⁹ to 10⁻⁴ m sized bubbles) grow as a result of pressure falling. Gases dissolved in the liquid diffuse into the bubble, filling and expand the cavity. When the liquid pressure in the constriction part (tubule) reaches less than or same as the liquid vapor pressure, the volume of the bubble will rapidly increase as the result of evaporation from the bubble’s surface. When the vapor-gas bubble moves into the increasing pressure zone of the venturi (outlet), condensation will occur followed by implosive size reduction of the bubble, thus leaving empty spaces around the bubble. These empty spaces will be filled by high speed liquid (with speed over 100 m/s), causing compression of the remaining gases locally. The
strongly compressed gas (over 100 MPa) will then undergo an expansion, which causes an explosion that rise the number of bubbles. During the explosion, due to the liquid’s inertia, the pressure drops and repeated evaporating occur on the surface of the growing bubble. This is repeated even hundred many times, causing the movement of cavitation zone beyond the constriction part.

Figure 2. Hydrodynamic Cavitations in Venturi

In the hydrodynamic cavitation process OH radicals and \( \text{H}_2\text{O}_2 \) are produced through the reactions as follow [16]:

\[
\begin{align*}
\text{HOH} & \xrightarrow{\text{variation}} \text{H}^* + \cdot\text{OH} \\
\text{H}^* + \text{O}_2 & \rightarrow \text{HOO}^* \\
\text{O}_2 & \rightarrow \text{O}^* + \text{O}^* \\
\text{HOO}^* + \text{HOO}^* & \rightarrow \text{H}_2\text{O}_2 + \text{O}_2 \\
\text{H}^* + \text{O}_2 & \rightarrow \text{HOO}^* \\
\cdot\text{OH} + \cdot\text{OH} & \rightarrow \text{H}_2\text{O}_2
\end{align*}
\]

3. Experimental

This research was conducted using ozonation, hydrodynamic cavitation, and combination of both methods to process the tofu wastewater. The tofu wastewater used in this research was taken from a home industry of tofu located in Jabodetabek. The treatment process of the tofu wastewater on each variation methods was brought for as long as 180 minutes, with sampling of temperature, pH of feed, and process effluent being taken on minute 30, 60, 120, and 180, also the sampling of ozone off gas on minute 180. The variations used in this research were dosage of ozone for ozonation method and flow rate (2, 3, and 4 LPM) for hydrodynamic cavitation method. Variation that produces the best result from each ozonation and hydrodynamic cavitation method would be used for combination method.

The independent variables used in this research were: flow rate which was configured by changing the opening of the pump outlet valve (2, 3, and 4 LPM) and dosage of ozone which was configured by changing the configuration of ozonator being used. The dependent variables being identified in this research were the degradation of the tofu wastewater’s BOD, COD, and TSS after the treatment process.

The main equipment used in this research were: a modified unit of commercial ozonator of X-Troy type CHS 212, a Mazzei type 384 injector venturi, glass reactor with volume capacity of 500 mL, and a Nocchi centrifugal pump. The experimental setup for this research is shown in Figure 3.
Materials used in this research were potassium iodide (KI) 0.1 M, sulphuric acid (H₂SO₄) 2 M, sodium thiosulphate (Na₂S₂O₃) 0.01 M, amylum indicator 2%, potassium permanganate (KMnO₄) 0.0001 M, sodium hydroxide (NaOH), and distilled water. All chemicals were provided from Merck with proanalysis (p.a.) grade, except sodium hydroxide (technical grade).

4. Results and Discussion

4.1. Productivity Test of Ozonator

This test was done in order to identify the concentration of ozone (O₃) being produced from each configuration of ozonator used in this research, namely: configuration A, B, parallel A-B, and series A-B. The results from this test would be used to choose the 2 configuration for the dosage of ozone variations in this research. This test was carried out by using a bubbler and iodometry titration method. The productivity results from this test are shown in Figure 4.

Figure 4 explains that the ozonator with configuration of series A-B produce the highest O₃ concentration. The configuration of series A-B has bigger air to O₃ conversion ratio due to its setting which makes the air from aerator being directed into two separate CD chambers (A and B) continuously. Therefore, the intensity of air exposure to the high voltage electricity is doubled and the concentration of O₃ being produced is higher. From the results of this test, it was decided that the ozonator with configuration A (as the single variant) and A-B series (as the series variant) would be used as dosage of O₃ variations.

4.2. Initial Characterization of Tofu Wastewater

This characterization was done in order to identify the initial pollution load, specifically its pH, COD, BOD, and TSS value, from the tofu wastewater used in this research. Other than that, this characterization was also done to evaluate the significance of pre-treatment using physical filtration.
(filter cloth 350 mesh) towards the initial pollution load of the tofu wastewater. The results from this initial characterization are shown in Table 1.

Table 1. Initial Characterization Results of Tofu Wastewater

| Parameters   | Without Filtration | With Filtration |
|--------------|---------------------|-----------------|
| pH           | 3.7                 | 3.7             |
| TSS (mg/L)   | 1816                | 1368            |
| COD (mg/L)   | 9649                | 7412            |
| BOD (mg/L)   | 339                 | 39              |

The results in Table 1 show that the pre-treatment using physical filtration capable to lower the initial pollution load of tofu wastewater quite significantly. It indicates that a portion of organic compounds in tofu wastewater is in suspended solid phase. Table 1 also shows that the tofu wastewater used in this research has a very small BOD to COD ratio, <0.2. It indicates the likely of tofu wastewater to contain toxic compounds that cannot be degraded naturally.

4.3. Quantification of OH Radicals

This quantification was done in order to quantify the OH radicals produced from each treatment methods used in this research. The treatment methods used in this research are ozonation ([O]) with dosage of O₃ variations (single and series variants), hydrodynamic cavitation ([HC]) with flow rate variations (2, 3, and 4 LPM variants), and combination ([HC/O]) with biggest variants of flowrate and dosage of O₃ (4 LPM flow rate and series variant of ozonator). This quantification was done by using distilled water with 3.7 pH, so that it resembles the tofu wastewater used in this research. The method used in quantifying the OH radicals is permanganometry titration using KMnO₄ 0.0001 M. The quantification results are shown in Figure 5.

![OH radicals production for all variation of configurations](image)

Figure 5. OH radicals production for all variation of configurations

According to the results in Figure 5, the all of treatment configurations produced an identical trend, where the concentration of OH radicals increases when the treatment duration increases. In [O] method, the series variant produced higher concentration of OH radicals than the single variant, 4.718 compared to 3.485 mg/L on the 180th minutes. It was a result of O₃ productivity difference between single and series variants, where the series variant has bigger O₃ productivity of 129 mg/h compared to 58 mg/h of single variant. The higher the concentration of O₃ involved in the system, the higher the concentration of OH radicals produced through Equation (7) [17].

\[
O_3 + H_2O \rightarrow 2 \cdot OH + O_2 \tag{7}
\]

In [HC] method, it was found that the concentration of OH radicals increasing along with increases in the flow rate being used. The high flow rate in the inlet part of the venturi causing a higher liquid velocity in tubule part, which according to Bernoulli Law will produce bigger pressure drop. Bigger pressure drop produces bigger cavitation effect, which positively affect the production of OH radicals. The [HC] method with 4 LPM flowrate produced the biggest concentration of OH radicals, 6.078 mg/L on the 180th minutes, followed by 3 and 2 LPM flowrate with OH radicals’ concentration of
5.27 and 4.548 mg/L accordingly. In [HC] method, the OH radicals are produced through the reaction of oxygen and water vapor thermal dissociation within the cavitation micro bubbles as shown in Equation 8 and Equation 9 [17].

\[
\begin{align*}
\text{H}_2\text{O} & \rightarrow \cdot\text{OH} + \text{H}\cdot \quad (8) \\
\text{O}_2 + 2 \text{H}_2\text{O} & \rightarrow 4 \cdot\text{OH} \quad (9)
\end{align*}
\]

[HC/O] method produces the biggest concentration of OH radicals amongst other methods, 8.67 mg/L on the 180th minutes. It was possible due to the increasing of O₃ and oxygen solubility in system by hydrodynamic cavitation, which increase the OH radicals produced from Equation 7 and Equation 8. Cavitation phenomenon from [HC] gives a turbulence effect which helps in the mixing process of O₃ and oxygen in the liquid phase of the system.

4.4. Treatment Process of Tofu Wastewater

The wastewater used in this treatment process is the tofu wastewater which had been diluted by using distilled water with wastewater to distilled water ratio of 1:6. The tofu wastewater was diluted in order to prevent the wastewater from turning into foams during the treatment. The treatment process was held for a total of 180 minutes, with sample being taken on minutes 30th, 60th, 120th, and 180th. The results from this treatment process, which describe the degradation rate of tofu wastewater’s COD and TSS from each treatment methods are shown in Figure 6, 7, and 8.

![Figure 6](image)

**Figure 6.** Degradation of COD (a) dan TSS (b) by Ozonation [O] Method (pH = 4.0, Flow rate = 2 LPM)

Figure 6 (a) and 6 (b) illustrate the degradation performance of COD and TSS from [O] method, where the single variant capable of degrading 92 mg/L COD and 153 mg/L TSS, while the series variant capable of degrading 136 mg/L COD and 233 mg/L TSS during 180 minutes of process. The series variant had better performance in degrading COD and TSS, which was caused by the use of higher concentration of O₃. The higher the concentration of O₃ being used, the more the oxidative species that are produced which will positively affect the degradation of COD and TSS in the wastewater. O₃ reacts with TSS by forming flocs which serves to absorb the colloid within the wastewater [18]. O₃ doesn’t destroy TSS, but only plays as a coagulant agent which helps in generating flocs of suspended solids. The generating of flocs will reduce the total TSS within the system. However, the overall performance of [O] method in degrading COD and TSS was still categorized as low. It was caused by the foams produced from the wastewater during the treatment, which act as a trap for O₃ in the system. The majority of the O₃ were trapped inside the foams, brought back to the reservoir, before finally released to the atmosphere.
Figure 7. Degradation of COD (a) dan TSS (b) by Hydrodynamic Cavitation [HC] Method (pH = 4.0)

Figure 7 (a) and 7 (b) illustrate the degradation performance of COD and TSS from [HC] method. The 4 LPM variant produced the best performance with degradation of 485 mg/L COD and 288 mg/L TSS, followed by 3 LPM variant with 239 mg/L COD and 275 mg/L TSS, and 2 LPM variant with 221 mg/L COD and 259 mg/L TSS during the 180 minutes of process. During the process, the cavitation micro bubbles from [HC] establishing a local extreme condition of pressure and temperature which initiate the thermal dissociation reaction of water as illustrated in Equation 1 to Equation 6. From these reactions, oxidative species such as OH radicals, hydrogen peroxide, and O$_3$ are produced [19]. The higher the flow rate being used, the stronger the cavitation phenomenon and the bigger the quantity of oxidative species being generated in the system. The cavitation phenomenon also gives an increase in contact area between oxidative species and pollutant compounds, thus causing a more effective degradation process [20]. The local effects of extreme thermal-mechanic from cavitation phenomenon are also involved in degrading the pollutant compounds. Those things made the degradation performance of the [HC] method better than [O] method.

Figure 8. Degradation of COD (a) dan TSS (b) by Combination [HC/O] Method (pH = 3.8, Flow rate = 4 LPM, Ozonator = Series Variant)

Figure 8 (a) and 8 (b) illustrate the degradation performance of COD and TSS from [HC/O] method, where this method was capable of degrading 943 mg/L COD and 293 mg/L TSS during 180 minutes of process. The result was even better than the sum of result from [HC] method 4 LPM variant and [O] method series variant separately. This result match the statement which said that [HC] and [O] method have a good synergy and could be combined to produce a more efficient oxidation process [8]. The chemical effect in this [HC/O] method was better than other methods, where the injected O$_3$ was effectively dissolved to the system with the help of cavitation micro bubbles. It made the quantity of O$_3$ molecules involved in [HC/O] method was bigger than other methods. The
characteristic of ozone as a selective oxidizer helped the process to degrade COD and TSS more accurate and effective. The high concentration of OH radicals in [HC/O] method also helped in degrading COD and TSS in the system.

![Figure 9. Degradation Performance from All Methods (pH = 3.7-4.0)](image)

Figure 9 illustrates the comparison of COD and TSS degradation performance between all of the treatment methods used in this research, namely [O], [HC], and [HC/O] method. The graph in Figure 9 shows that [HC/O] method produced the best degradation performance. It is consistent with the statement which said that [HC] has the capability to increase the saturation limit of a solution/system against the oxidizer gases and intensify the oxidation reaction through the increasing of contact area between oxidizer and pollutant [15]. Because of that increase in the system’s saturation limit, the quantity of O₃ gas dissolved in the system became bigger. The increase in oxidizer-pollutant contact area also made the oxidation took place in the system became more intensive and effective.

5. Conclusions
The [O], [HC], and [HC/O] method are proven to have capability in degrading pollutant parameters of tofu wastewater, where the [HC/O] method (4 LPM variant of flowrate and series variant of ozonator) produced the best result with degradation of 943 mg/L COD and 293 mg/L TSS in 180 minutes duration of process.

The flowrate being used has a positive impact on the COD and TSS degradation performance, where the higher the flowrate then the better the degradation performance. The 4 LPM variant of [HC] was found capable of degrading 485 mg/L COD and 288 mg/L TSS, while the 3 and 2 LPM variants were only capable of degrading 239 mg/L COD and 275 mg/L TSS, 221 mg/L COD and 259 mg/L TSS accordingly.

The dosage of ozone being used has a positive impact on the COD and TSS degradation performance, where the higher the dosage of ozone then the higher the degradation performance. The series variant of [O] method (128.92 mg/h O₃) was found capable of degrading 136 mg/L COD and 233 mg/L TSS, while the single variant (58.04 mg/h O₃) was only capable of degrading 92 mg/L COD and 153 mg/L TSS accordingly.

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