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PAPER

Analysis and characterization of enhanced kinetic reaction on ozone generation using negative corona discharge

Fuei Pien Chee[1] , Nur Syahirah Zainuddin[1], Howe Ling Ha[2] and Jedol Dayou[1]

[1] Faculty of Science and Natural Resources, University of Malaysia Sabah, 88400 Kota Kinabalu, Sabah, Malaysia
[2] School of Engineering, University of Hull, Hull, HU6 7RX, United Kingdom

E-mail: fpchee06@gmail.com

Abstract

Background and objective: Ozone gas has been used in microorganism treatment and is known as one of the strongest oxidizing agents. The main limitation of ozone is decay rapidly due to its short halflife. In this study, a novel method was established to enhance the production of ozonated water with lower decomposition time. *Materials and method:* The newly developed prototype was designed with improved technology to increase the kinetic reaction between ozone and water utilizing the parameters of high pressure and continuous flow. Ozonated water produced was examined using dissolved ozone meter with maximum detection of 20 mg L^{-1}. The reaction and decomposition modeling of this system was developed and the dynamic structure of produced ozonated water was investigated using Raman spectroscopy. *Results:* The findings showed that the newly developed prototype facilitates a higher concentration of ozonated water with an ozone transfer rate of 2.5 L min^{-1} and a lower decomposition rate with an average half-life 200 min. The results obtained from Raman spectroscopy showed the increases in ozonated water concentration leading to a decrease in the intensity of Raman peak, more likely approaching the Raman intensity of distilled water. *Conclusion:* This system was proven to be efficient in producing ozonated water at high volume and concentration with longer half-life which suits the domestic and industrial application requirements.

1. Introduction

Ozone is a naturally occurring gas in the atmosphere and is known as trioxygent. Each ozone molecule contains three atoms of oxygen. Ozone is characterized as an inorganic molecule with a chemical formula of O$_3$. In general, there are several methods for producing ozone gas which is by using Corona Discharge (CD), Dielectric Barrier Discharge (DBD), Atmospheric Pressure Glow Discharge (APGD) and Ultraviolet Ray (UV) [1–5]. In either case, the principle behind the production of ozone involves the oxygen bond breaking by applying energy to form a single oxygen atom which combines with other oxygen molecules to produce ozone [1–5].

There were many kinds of research that had been conducted on ozone and is found useful in microorganism treatment such as sterilization, water treatment, food preservation, product storage, odor control, health medicine, etc. Even though it is known as a toxic gas but under a limited range and control environment, it has been used widely in industries [6, 7].

Ozone had been known as the strongest commercially available oxidizing agent that can be produced, however, the ozone molecule is unstable and cannot be stored for future use. For this reason, ozone must be generated close to the point of the application then used immediately [6–10]. Besides that, the existing ozone system passes dry, clean air through a high voltage electric discharge to produce ozone of approximately 1 percent or 10 000 mg Lmg L^{-1}. However, the ozone produced decay rapidly as ozone is unstable with a short half-life. Another option is by dissolving ozone into the water before the treatment. However, this approach is still under investigation as the outcome is inadequate. The main factor for the unsatisfactory result is because of low ozone mass transfer rate into water. Nevertheless, current technology is still unable to confine ozone in water for a long period and therefore the half-life is short [11, 12]. As a result, sophisticated equipment and operational

Figure 1. Dissolved ozone analyzer KRDRY-2056.

cost are the main issues faced by most applications on ozone treatment particularly in sterilizing, disinfection and microbe removal. The issue of the short half-life of ozone can be solved by dissolving ozone into water as confinement media for later application. This can be done by improving the diffusion efficiency of ozone in water including increasing the ozone concentration in the air, increasing both air and water pressure, decreasing the water temperature and providing higher probability for collisional activities between the ozone and water particles [13, 14].

The objective of the research was to establish the effectiveness of the developed prototype in producing ozonated water with good qualities, specifically with a longer half-life and higher concentrations. The ozonated water produced by the system is an ideal solution for any treatment without owning the whole ozonation system. Hence, the results of characterize ozonated water using Raman spectroscopy is to allow interpretation of the structure and molecular dynamics in various concentrations.

2. Materials and methods

2.1. Experimental set-up

This research was carried out for two years starting in Febuary 2016. The experiment was conducted in the Laboratory at the Faculty of Science and Natural Resources, University Malaysia Sabah, Malaysia.

In this study, the dissolved ozonated water concentration was measured using ozone analyzer KRDRY-2056 (Hebei Create Instrumentation Technologies Co., Ltd) as shown in figure 1. This analyzer displays several parameters at the same time which are dissolved ozone value, temperature value, and current. The measuring range of this ozone analyzer for dissolved ozone value is 0–20 mg L^{-1} (precision of ± 0.01 mg L^{-1}) which corresponds to 4–20 mA current output and for temperature value is 0 °C to 60 °C (precision of ± 0.5 °C).

$$\text{Output current (mA): } I = 16 \times (C - A)/(B - A) + 4 \tag{1}$$

Where I is current output, C for instrument current measuring dissolved ozone, value A for setting of 4 mA and B for setting of 20 mA.

2.2. Study design

A system was developed to produce an ozone water system with good qualities for any treatment also enhances the scopes of application of ozonated water treatment on a larger scale, and allows this treatment on more applications. All the chemical and reagent used in this research was laboratory grade. The entire system flow was divided into three main phases. The first phase is ozone gas production, the second phase is ozone water production and the third phase is ozone water storage.

2.2.1. Ozone generation using corona discharge

The first phase of the system is ozone gas production. Corona discharge method was applied to generate ozone gas in this research [15, 16]. The corona discharges (CD) are used to produce charged particle. The working principle of CD is by passing air through a high voltage electric field, which is close to ignition voltage required for electric breakdown. The air or concentrated oxygen (O_2) passed through the corona containing free electron
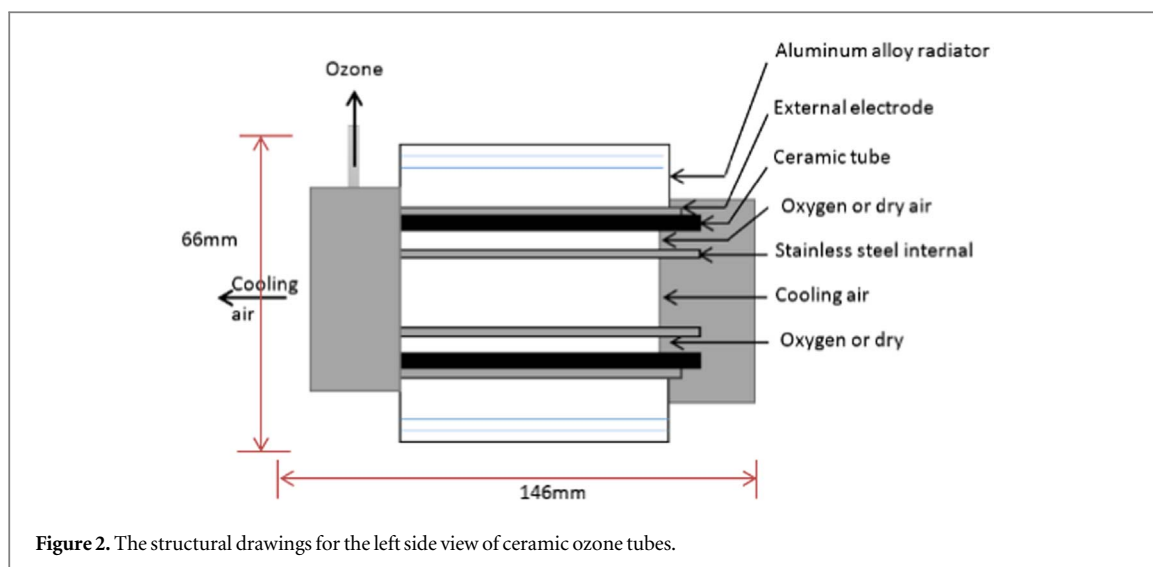
Figure 2. The structural drawings for the left side view of ceramic ozone tubes.

which causes the oxygen bond to split and produce free radicals (O^-) and combine with other O_2 molecules to create Ozone (O_3). The plasma electron provides the energy that overcomes binding energy of O_2 into O is 5.13 eV with the cross section around 1.3×10^{-18} cm^2 as shown in equation (2). O^-, thereupon will join with other O_2 to form O_3 with binding energy of 1.49 eV from background corona and cross sections of 3.7×10^{-17} cm^2 as shown in equation (2) [1].

$$O_2 \leftrightarrow O^- + O^- \tag{2}$$

$$O_2 + O^- \leftrightarrow O_3 \tag{3}$$
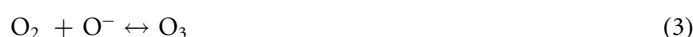
The type of corona used for ozone generation in this research was a ceramic ozone tube with dry air or oxygen-filled short gaps. Figure 2 shows the internal structure of the ozone tube. Aluminum alloy radiator was used for the wall of the tube while the end caps of ozone and ozone outlet were made of polytetrafluoroethylene which has high-purity corrosion-resistant fluorine rubber seal rings. Sheets of electrode comprise the second (discharge) layer was placed between the aluminum alloy and ceramic tube. The inner (dielectric) layer of each electrode was tubes of ceramic with high dielectric constant and a relative permittivity of 5.50. The stainless steel electrode was used as an internal electrode for both layers. The air cooling was used for internal and external electrodes. The tube was designed with a parallel arrangement with a length of 146 mm and a width of 66 mm and also with a narrow discharge gap spacing.

Corona discharge method produces ozone by passing air through a high voltage electrical discharge. Then, the energy within the discharge converts a part of the oxygen to ozone. This ozone generator has an ozone capacity of 3 g h^{-1} with a concentration ratio of 0 to 20 gr m^{-3}. The energy use for this ozone generator is 115watts and the oxygen feed flow rate at 8LPM. The ozone is produced when oxygen molecules from the oxygen tank are dissociated by energy sources into oxygen atoms and subsequently collide with an oxygen molecule.

Air or pure oxygen could be used as the feed gas source and passes through the ozone generator. Using a feed gas source from outside air requires an air compressor to absorb enough air into the ozone generator, however by using pure oxygen, the system could be directly connected to the ozone generator. The gas stream from outside contains about 0.5% to 3% ozone by weight, whereas pure oxygen will form approximately two to four times that concentration. The commercial-grade oxygen from the oxygen tank was used as the gas source at 68950 Pascal constant pressure for each experiment. This amount of oxygen pressure was used as it is the optimum oxygen pressure that able to give a high ozonated water concentration for the reaction. In this study, the designed ozone generating system was installed in a clean and dry area with good ventilation. It was placed above the water tank level to avoid water travel down the ozone output hose and entering the ozone generator when the pressure lost in the venturi vacuum.

2.2.2. Production of ozonated water using venturi injection technique

The second phase proceeded when the ozone gas flowed into the water stream via the suction port in the injector and dissolved with water. The venturi injection dissolved the ozone gas in water by pressure. The pressure creates constriction in the fluid flow and causing suction of ozone. The venturi injector is used to dissolve ozone in water. By using a venturi injector the ozone mass transfer transfers rate is raised to 98 percent. Then, the
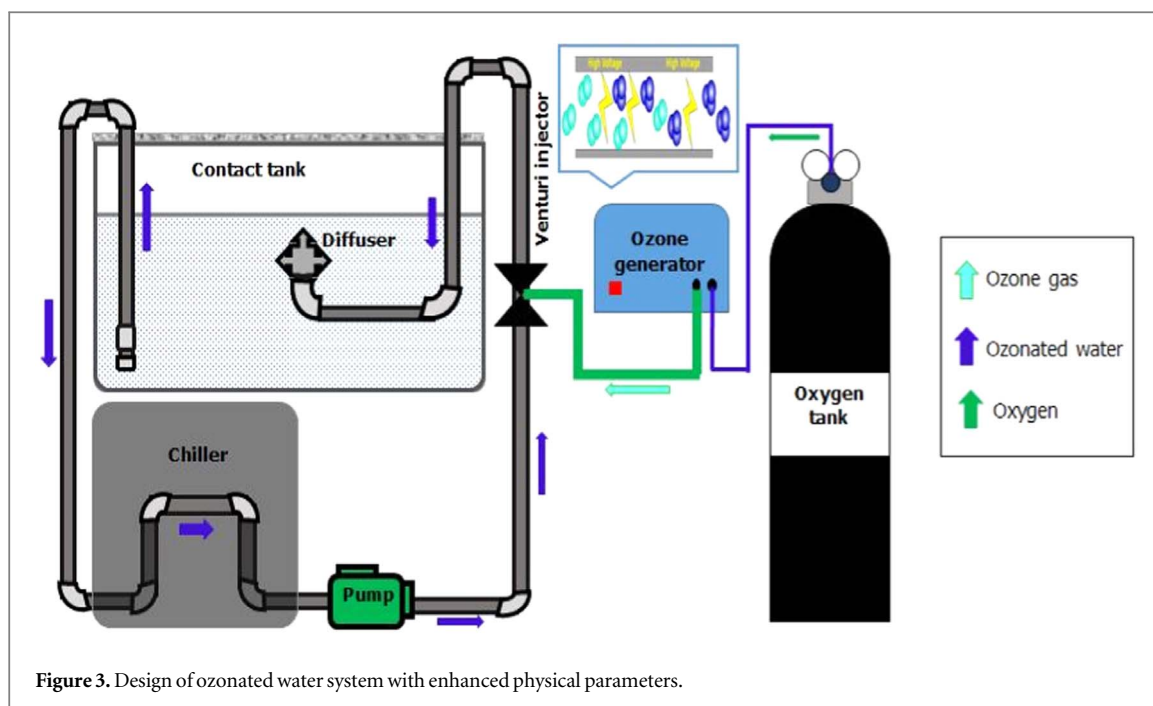
Figure 3. Design of ozonated water system with enhanced physical parameters.

dissolved ozone was added into the ozone water chamber and then passed through to the chiller. The chiller was used to reduce the contact temperature and therefore increase the half-life of ozonated water. This chiller has a maximum power of 600 Watt and can reduce to a minimum temperature of 5 Celsius. Then, the ozonated water will flow into the water pump (Model QB-60, ATAS.com) which increases the water pressure. This water pump has a power of 0.5 HP and inclines the water speed at about 2850 RPM. The increment of water pressure is the main concern in this system as it could increase the mass of ozone transfer in water, thus increasing the ozonated water concentration ozonated water. Next, ozonated water from the water pump was vented back into the venturi injector and dissolved again with ozone gas. This process flowed continuously until the solution of dissolved ozone reached the desired concentration. The water pump also serves the function to regulate ozonated water in the system to ensure that the ozonated water reaches the required concentration. The ozonated water was stored in a transparent contact tank that gives a visible vision of the overall process. This allowed easier inspection of the condition inside the tank such as the presence and size of ozone bubbles during the reaction. The contact tank was also designed with a glass panel that covers the top part of the contactor to prevent the escape of ozone gas. Figure 3 shows the system design.

The third phase was the storage of ozonated water which would be applied for the ozonation process. The required concentration of ozonated water can be applied for several treatments. The ozonated water can be effectively used to destroy viruses and bacteria and the process utilizes a short contact time approximately 10 to 30 min. Besides that, there is no harmful residual factor that needs to be removed after ozonation because ozone decomposed rapidly.

2.3. Characterization of ozonated water sample using raman spectroscopy

In order to analyse changes in composition of the water, the Raman spectroscopy was used for different samples of ozonated water concentration to test for impurities and the presence of foreign molecules. In this experiment, the HR1-T 532 nm, 200 mW Low noise laser Raman spectrometers were used in identifying organic compounds of ozonated water. The ASEQ RM-1 Raman spectrometer was pre-validated by polypropylene testing. This spectrometer measures spectral range from approximately 200 to 4510 cm^{-1} with spectra resolution of 8.5 cm^{-1}.

The Raman shift was recorded by first running a background reading an empty cuvette to remove noise from the final reading. Next, the cuvette was filled with the ozonated-water and another Raman reading was recorded. To calculate the Raman shift, the ozonated water intensity spectrum was divided by the background spectrum, yielding the Raman shift of the ozonated water.

The characterization using Raman Spectroscopy was conducted for the concentration of ozonated water at 5 mg L^{-1}, 10 mg L^{-1}, 15 mg L^{-1}, and 20 mg L^{-1}. The Raman spectrum of the ozonated water after decomposed was also studied. All the findings were compared with the spectra for distilled water and tap water.
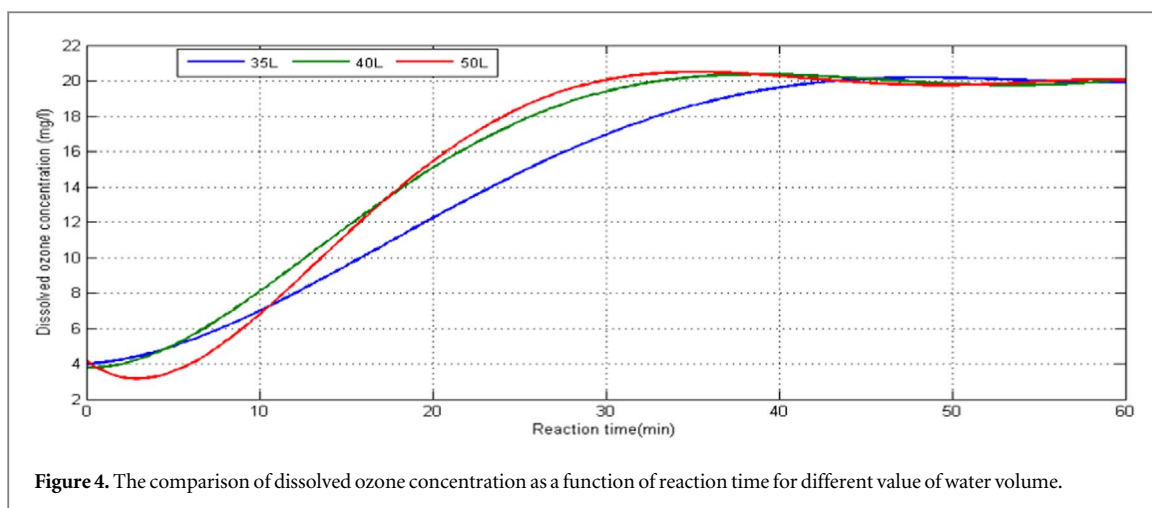
Figure 4. The comparison of dissolved ozone concentration as a function of reaction time for different value of water volume.
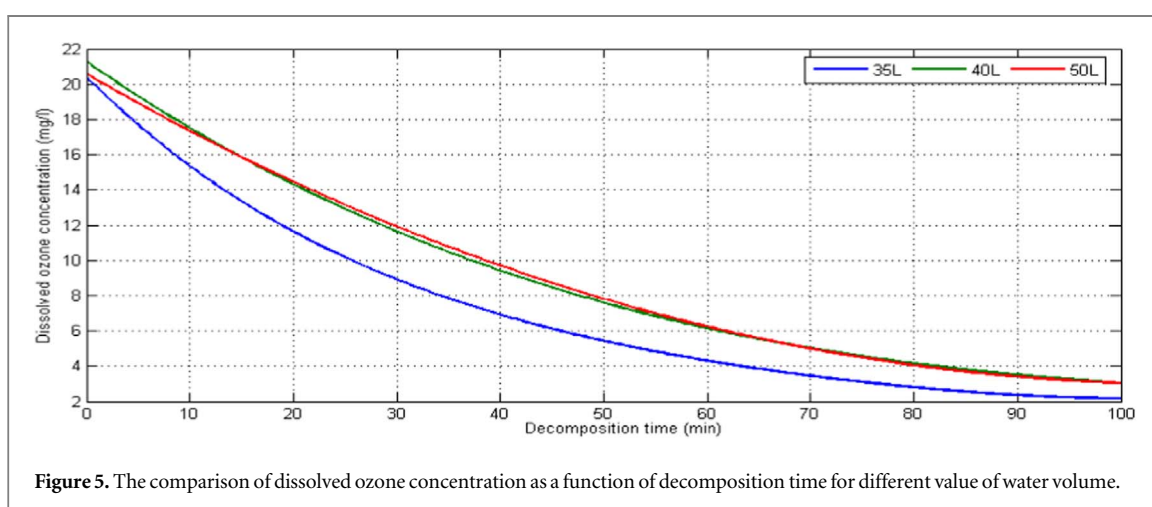

Figure 5. The comparison of dissolved ozone concentration as a function of decomposition time for different value of water volume.

3. Results

3.1. Parameter and quality testing of ozonated water

All experimental data were analyzed using Matlab Software R2010a. Figures 4 and 5 establish the relationship of ozonated water concentration using developed prototype at the different volumes of water which is 35 L, 40 L, and 50 L.

From figure 4, the dissolved ozone concentration for all volume of water steadily inclined up to 20 mg L^{-1}. The ozonated water concentration at 35 L, 40 L, and 50 L reached the maximum concentration at 40 min, 30 min and 25 min respectively.
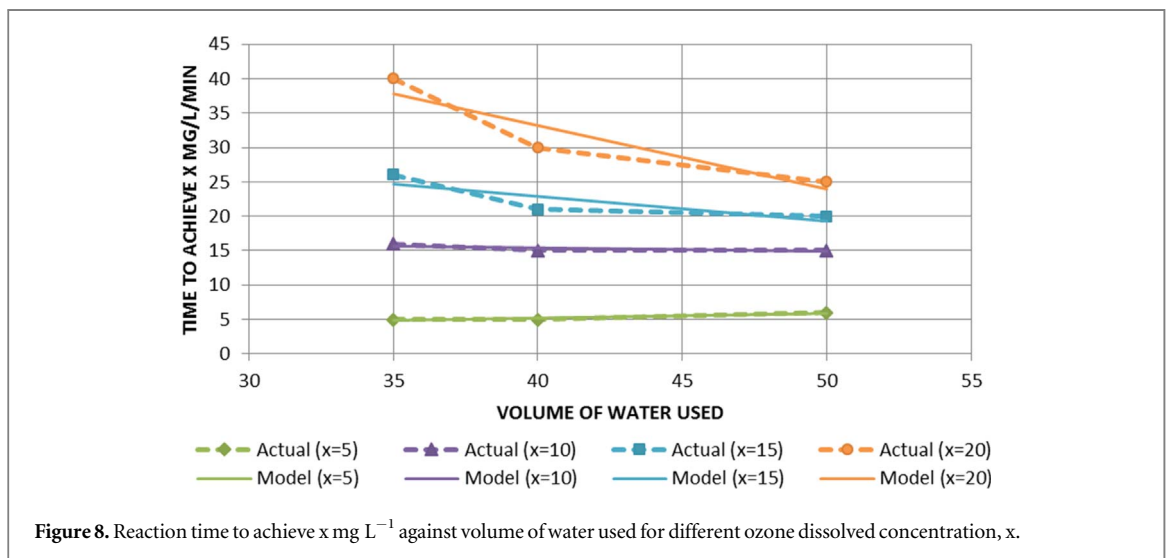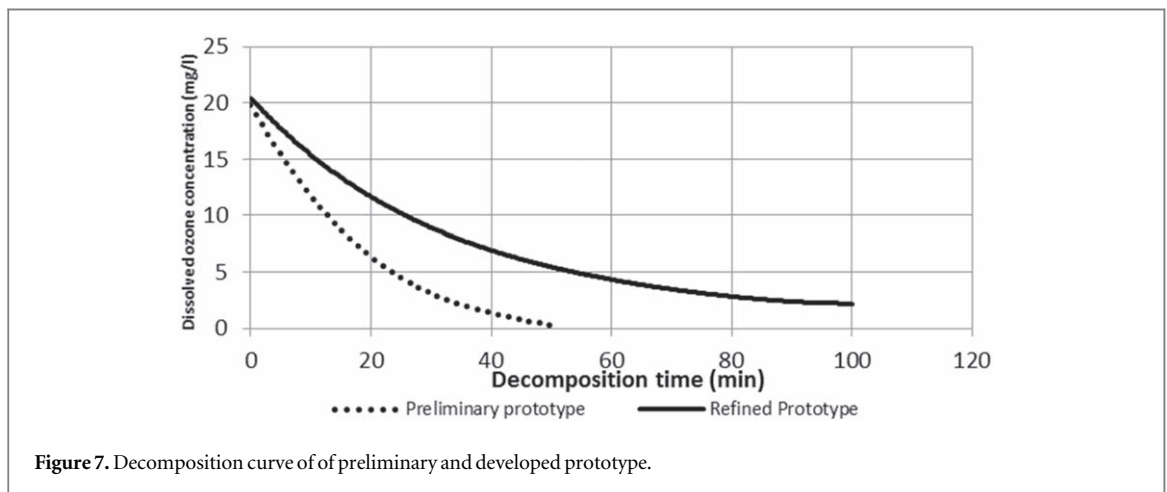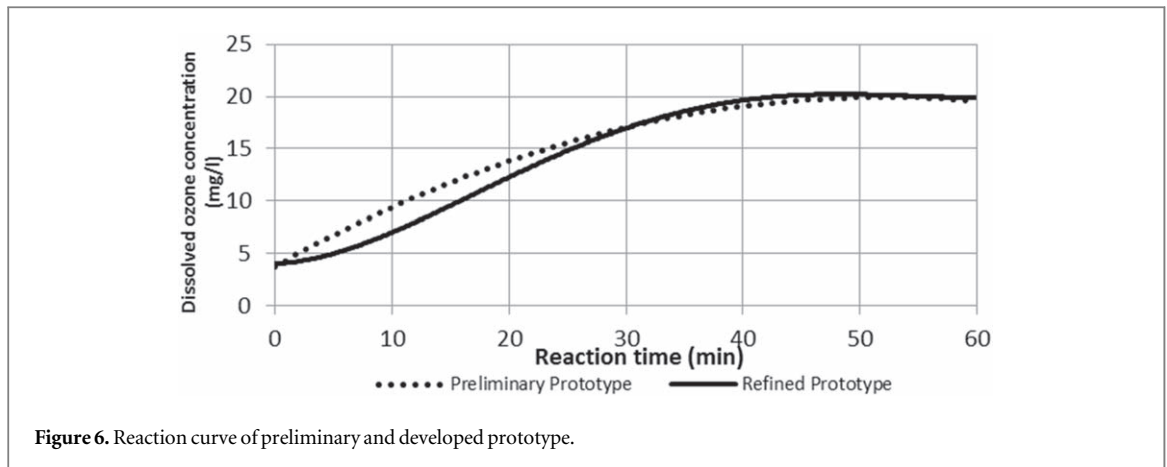
From figure 5, the decomposition time of ozonated water using a developed prototype shows a gradually decreasing trend over 100 min for all tested volume of ozonated water. The rate of decomposition of ozone for 35 L of water is highest compared to those of 40 L and 50 L of water.

The data was then compared between the system proposed in this paper (developed prototype) and our first design [15] (preliminary prototype). Figures 6 and 7 shows the comparisons between the preliminary prototype [15] and the developed prototype.

Figure 6 reveals that there is no significant difference in reaction time between these two prototypes. However, as shown in figure 7, the decomposition time of ozonated water from the preliminary prototype is shorter than the developed prototype. The decomposition rate of ozonated water produced by the preliminary prototype is 2 times faster than that by developed prototype, which occurs at 50 min for first and 100 min for the latter.

3.2. Reaction and decomposition modeling of developed prototype

To deduce the relationship between volume of water used and time (reaction time and decomposition time), figures 8 and 9 are plotted.

Figure 6. Reaction curve of preliminary and developed prototype.

Figure 7. Decomposition curve of of preliminary and developed prototype.

Figure 8. Reaction time to achieve x mg L^{-1} against volume of water used for different ozone dissolved concentration, x.

For both reaction time and decomposition time, all the parameters such as time and volume of water can be related in a straight line equation,

$$t = mV + k \qquad (4)$$

Where t = time, m = gradient of line, V = volume of water and k = y intercept of line.

For reaction time as illustrated in figure 7, $_{Mr}$ varies inversely with the ozone dissolved concentration achieved at time t while k_r varies accordingly to the ozone dissolved concentration achieved at time t. Through calculation, it is found that m_r relates to ozone dissolved concentration by the equation;

Figure 9. Decomposition time to achieve x mg L^{-1} against volume of water used for different ozone dissolved concentration, x.
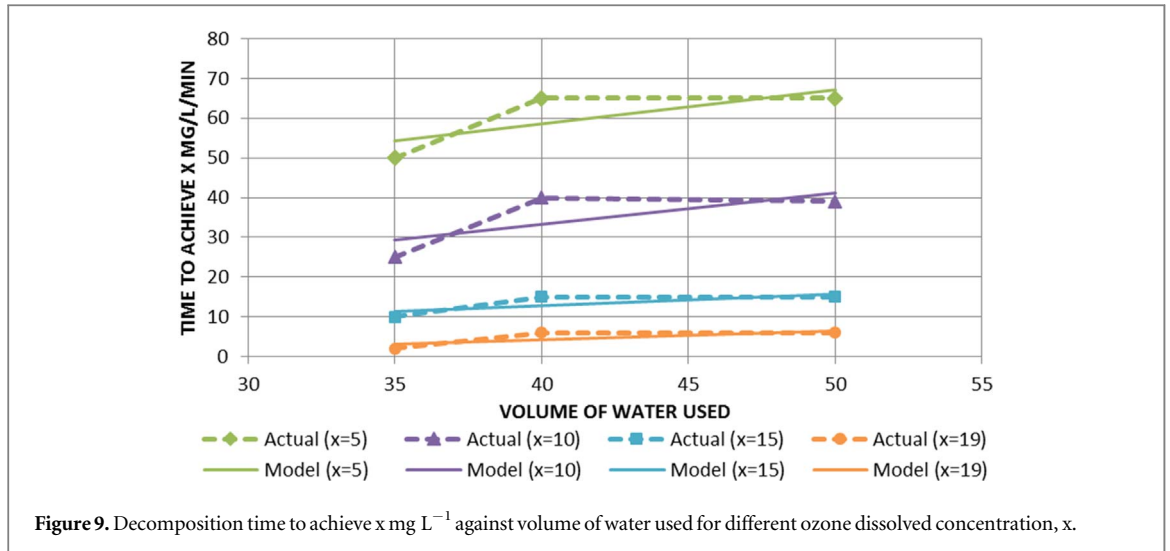
$$m_r = -0.066x + 0.5072 \tag{5}$$

Where x = ozone dissolved concentration at time t. Meanwhile, k_r relates to x by the equation,

$$k_r = 4.47x - 23.964 \tag{6}$$

Substitution both m_r and k_r into the equation,

$$t_r = m_r V + k_r \tag{7}$$

The following equation is obtained for the reaction model of this prototype;

$$t_r = (-0.066x + 0.5072)V + 4.47x - 23.964 \tag{8}$$

This equation is valid when $t \neq 0$ and until the time $x_{\max}$ is achieved.

For decomposition time, m_d is inversely proportional to the dissolved ozone concentration achieved at time t, x and k_d is directly proportional to the x. Through the calculation, m_d can be said to relate to x by equation (6), the calculation was started at x = 19 which is after the decomposition activities started.

$$m_d = -0.051x + 1.1646 \tag{9}$$

Meanwhile, k_d relates to x by equation,

$$k_d = -1.8896x + 28.844 \tag{10}$$

Substitution both m_d and m_k into the equation,

$$t_d = m_d V + k_d \tag{11}$$

The following equation is obtained for the decomposition model of this prototype;

$$t_d = (-0.051x + 1.1626)V - 1.8896x + 28.844 \tag{12}$$

The equation also assumes that at $t = 0$, $x = x_{\max} = 20$ mg l^{-1}.

3.3. Characterization of ozonated water

Ozonated water that was produced from the developed prototype will be characterized using Raman spectroscopy. This test is to detect the spectrum and wavelength of the ozonated water. The Raman scattering technique is vibrational molecule spectroscopy which derives from the light scattering process by using the laser. During the process, a laser photon is scattered by a sample molecule and loses or gains energy. The amount of energy loss is seen as a change in wavelength of the irradiation photon. This energy loss is characteristic of a particular bond in the molecule.

The comparison of Raman Spectrum between distilled water and tap water was shown in figure 10. This analysis characterizes internal molecular vibration with the peaks in the range of 0 cm^{-1} to 4500 cm^{-1}. The major peaks of the distilled water spectrum are observed at 200 cm^{-1}, 1000 cm^{-1}, and 3500 cm^{-1}. An intense band of distilled waster is observed at Raman shift approximately 3500 cm^{-1} to 4000 cm^{-1} with the absorbance of 35. Six major peaks were observed from the Raman spectra of tap water which are at Raman shift of 200 cm^{-1}, 400 cm^{-1}, 1000 cm^{-1}, 2800 cm^{-1}, 3300 cm^{-1} and 3500 cm^{-1}.

Figure 11 shows the Raman spectral for ozonated water at various concentrations compared with distilled water. All the spectra show similarity on Raman shift but pronounce a significant difference in the Raman intensity. The graph shows that as concentration increases, the intensity of the peaks decreases. The ozonated
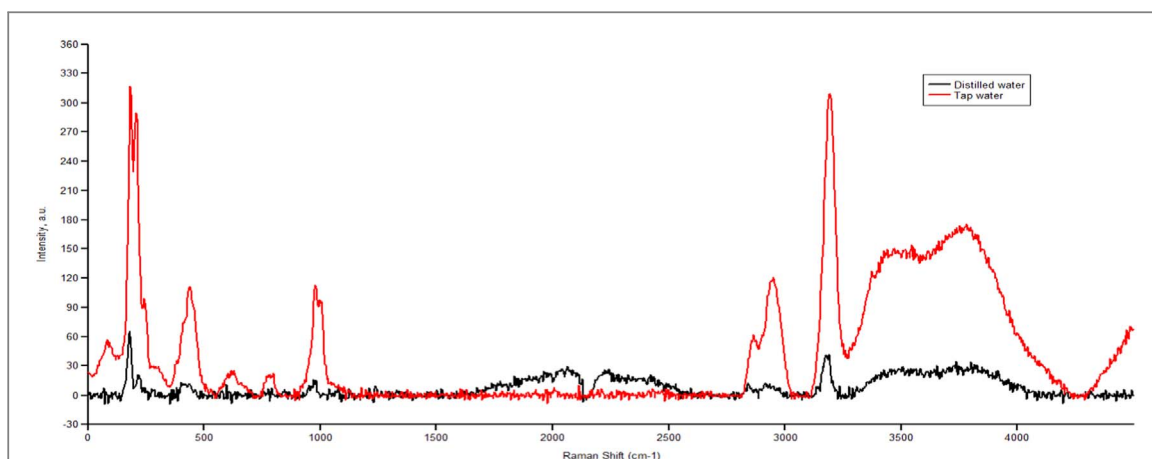
Figure 10. Comparison of distilled water Raman intensities with tap water sample at room temperature.
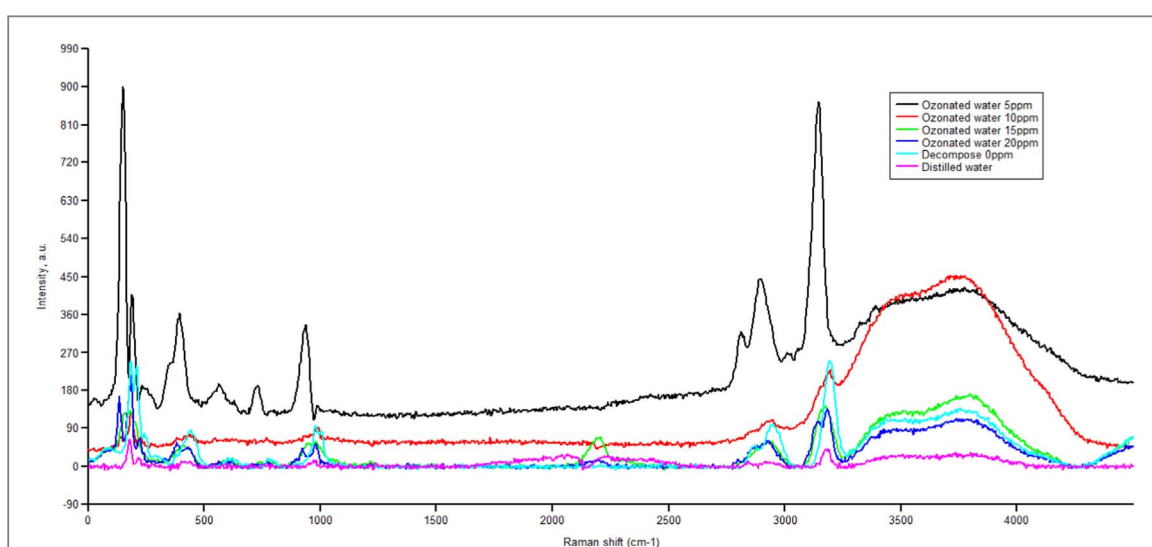

Figure 11. Comparison of Raman spectral of ozonated water at different PPM with distilled water at room temperature.

water at 5 ppm shows the highest peaks which are approximately 900 absorbent. The most intense peaks are 200 cm^{-1}, 400 cm^{-1}, 1000 cm^{-1}, 2800 cm^{-1}, 3300 cm^{-1} and 3500 cm^{-1} for all samples. Moreover, the graph reveals with great precision the complex spectra shape by six Gaussian-shaped constituents at positions approximately 3500 cm^{-1}. These six constituents were shown in figure 12.

4. Discussion

From the relationship of the result of ozonated water concentration using developed prototype at the different volume of water (figure 4), for each volume of water; the ozonated water concentration begins at around 3.5 mg L^{-1}. These initial values of dissolved ozone concentration were considered as tap water value due to the chlorine effect and some impurities detection by the ozone analyzer. In general, the reaction time for each volume of water shows an increasing trend and achieved a maximum ozone concentration of 20 mg L^{-1}. This result is considered as the highers ozone concentration since the measuring range of ozone analyzer (KRDRY-2056; Hebei Create Instrumentation Technologies Co., Ltd) for dissolved ozone value is 0–20 ml L^{-1} (precision of ±0.01 mg L^{-1}). Even though having a similar trend, it shows a clear distinction that the reaction time to achieve maximum dissolved ozone concentration decreases as the volume of water increases. The data indicates the reaction time for 50 L of water to reach maximum ozone dissolved concentration is the shortest which is approximately 25 min. This proves that ozone bubbles are difficult to escape from the water surface when the water level is high, thus making the rate of escape of ozone bubbles from water surface relatively lower than the rate of mixing of ozone and water at the venturi injector. The result obtained is showing similarities with the
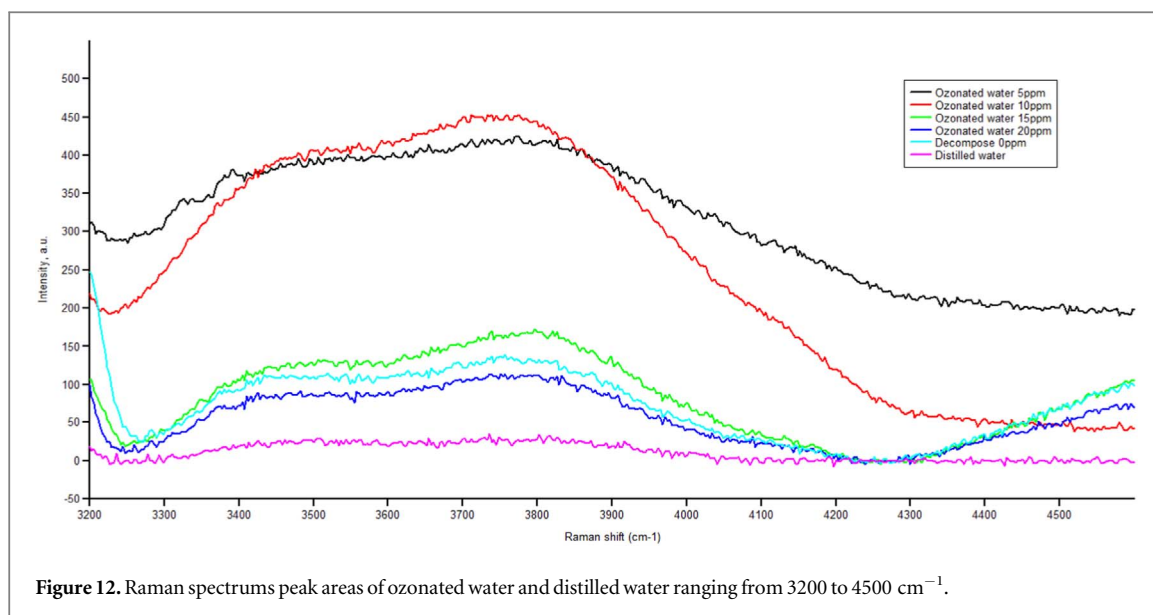
Figure 12. Raman spectrums peak areas of ozonated water and distilled water ranging from 3200 to 4500 cm^{-1}.

findings from [12] stated that the kinetic of ozone decomposition in water is initiated by molecule in the water itself which are H_2O, OH^-, and H_3O^+.

From the result of the decomposition time of ozonated water using the developed prototype at the different volume of water (figure 5) show a gradually decreasing over a period of 100 min. This indicates that the ozone concentration in the ozonated water is approaching the value of distilled water which is approximately zero after decompose. This is due to the disinfecting nature of ozone in water. The data proves to be evidence that ozone can destroy viruses and bacteria, and reduce the concentration of iron, manganese, and sulfur in the water. This shows that ozone can be a good substitution for chlorine in water treatment. The rate of decomposition of ozone for 35 L of water is highest compared to those of 40 L and 50 L of water. This may be due to the lower level of water height that allows the easier escape of ozone gas from the water surface. When the contact tank is confined with a higher volume; the escape activity of ozone is slower [12]. Since this was a temperature independence experiment, the half-life of ozonated water will, therefore, establish its standard properties at a room temperature condition. This occurs when the escape rate of the ozone in the ozonated water is identical.

The results were then compared for 35 L volume of water between previous research preliminary prototype [15] with the developed prototype as design in this research. The patent of the graph for the reaction time is standardized for both prototypes (figure 6). For decomposition time, the dissolved ozone concentration for both prototypes varies inversely with the decomposition time (figure 7). The result reveals that the half-life of ozonated water produced using the developed prototype is two times longer than the ozonated water produced from preliminary prototype [15]. This finding proved that the developed prototype greatly improves the ozonated water half-life.

In this research, it was found that the reaction equation of this prototype (figure 8) is $t_r = (-0.066x + 0.5072)V + 4.47x - 23.964$, where the equation is valid when $t \neq 0$ and until the time x_{max} is achieved. Moreover, for the decomposition reaction (figure 9) is $t_d = (-0.051x + 1.1626)V - 1.8896x + 28.844$ where the equation was assumes that at $t = 0$, $x = x_{max} = 20$ mg l^{-1}.

The comparison of Raman Spectra between distilled water and tap water was shown in figure 10. Both spectra have similar Raman shifts but showing a significant difference in Raman intensity. The presence of intense peaks indicates that high impurities content present in tap water. Tap water and distilled water are very similar in content. The peak at 1000 cm^{-1} corresponds to the presence of ^{v}(C=S) vibrations. Furthermore, the peak at 2800, 3100 and 3500 cm^{-1} are assigned to the stretching of ^{v}(C–H), ^{v}(=(C–H)) and a hydroxyl group ^{v}(O–H) respectively [5, 17]. The intensity of distilled water is very much lower compared to tap water. The difference between OH stretching intensity of tap water and distilled water is about 2.7 times stronger. According to [18], the reduced Raman spectrum in the O–H vibration region of tap water are well explained by a temporal around oxygen atom and not a single H_2O molecule. Moreover, the intense band at the Raman shift approximately 3500 cm^{-1} to 4000 cm^{-1} is originating from aliphatic chain vibrations ^{v}(O–H) which is classified as OH stretching [17]. This result was supported by Paulo-SP, 2017, who stated that the broadband in the OH stretching in the region 2700 cm^{-1} to 4000 cm^{-1} which can be considered as a continuous distribution of hydrogen bond concerning both energetic and geometric parameters [19]. Besides that, this distribution is temperature dependent, since a typical hydrogen bond becomes weaker at the higher temperature.

Identification of Raman spectra at different concentration ozonated water (figure 11) shows that the intensity of the peak decreases as the ozonated water concentration increases. The lowest concentration of ozonated water shows the highest peaks. This can be explained by the presence of impurities in the tap water which not efficiently treated due to inadequate of ozone concentration.

Moreover, the complex spectra by six Gaussian-shaped (figure 12) probably originating from the region $3500\ cm^{-1}$. The peaks of $3500\ cm^{-1}$ Raman shift correspond to the presence of the OH stretching hydroxyl group. These peaks show the most interest area where the large spike occurs in the range around $3300\ cm^{-1}$ to $4000\ cm^{-1}$. It is clear that a high concentration of ozonated water results in the appearance of indistinct and broad Gaussian-shape OH stretching which likely equal to distilled water. This result support, ozone as a strong oxidant; it will react with various oxidizable inorganic and organic materials. The reaction with unsaturated organic compounds gives unstable ozonides, which can decompose violently [12–21]. Besides that, the lower line of OH stretching of 20 ppm ozonated water due to the ozone was distributed equally and fully dissolved in water. Meaning that the tap water was completely treated by ozone leading to the nearest spectrum to distilled water. The shift in OH stretching towards higher absorbance, as ozonated water concentration decreases. The 5 ppm and 10 ppm of ozonated water show the highest OH peak intensity, followed by 15 ppm and 20 ppm. When ozone decomposes in water, the free radicals hydrogen peroxy (HO_2) and hydroxyl (OH) that are formed play an active role in the disinfection process [22]. The 0 ppm of ozonated water spectrum line was closed with distilled water and almost similar with 20 ppm ozonated water. Meaning that even ozone is fully decomposed, the water is cleared from the existence of additive impurities. This proved that ozonated water can be categorizing as a powerful oxidizing agent and also can be used in water treatment.

5. Conclusion

This study proposed an enhancement of ozonated water system, with corona discharge ozone generator and venturi injection method, and the process performance and product quality were evaluated. The developed prototype with enhancement of reaction kinetic has archived ozonated water concentration 20 mg L^{-1} at 40, 30 and 25 min for volume of 35, 40 and 50 liter respectively. For decomposition time it takes approximately 100 min to reach the concentration at 0 mg L^{-1}. High concentration of ozonated water results in the appearance of indistinct and broad Gaussian-shape of OH stretching which likely equal to distilled water. The increases in ozonated water concentration leading to a decrease in the intensity of Raman peak, more likely approaching to the Raman intensity of distilled water.

The ozonated water produced can be used for *ex situ* treatment that can be beneficial for many used. Treatment can be done easily by layman using ozonated water. Thus, operational cost, skill, and safety issue can be eliminated. This study will help researcher to uncover the critical areas of ozonated water half- life and characterization. Thus a new theory on ozonated water reaction and decompostion rate may be arrived at.

ORCID iDs

Fuei Pien Chee ⓘ https://orcid.org/0000-0002-9782-5572

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