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Treatment of benzene-contaminated groundwater by ozone micro-nano-bubbles

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ABSTRACT

Benzene, a kind of volatile organic compound (VOCs) in organics-contaminated groundwater, is harmful to public health and the environment. Traditional methods of groundwater remediation such as pump & treat and chemical oxidation have limitations of high cost, low time efficiency, and secondary pollution. It is important to develop green and sustainable in-situ groundwater remediation technology. Ozone micro-nano-bubble (OMNB) technology, with a high transfer efficiency of oxidant, strong oxidation capacity, and low secondary pollution, has great potential for in-situ remediation of organics-contaminated groundwater. In this study, benzene is treated with OMNBs. The effects of ozone and H_2O_2 dosage, CO_3^{2-}, fulvic acid and Cl^- concentrations, and pH on the treatment of benzene-containing wastewater are investigated. The results show that the micro-nano-bubbles (MNBs) greatly prolong the half-life of ozone and the benzene removal rate increases with increasing ozone dose. An optimal hydrogen peroxide to ozone molar ratio of 0.15 exists for the enhancement of hydrogen peroxide to benzene treatment by ozone MNBs. Carbonate and fulvic acid in water inhibit the degradation of benzene by depleting the oxidant in the reaction system, while chloride ions have a facilitative effect on benzene degradation. The best removal effect for benzene is obtained when the solution is weakly acidic and the removal rate decreases with increasing pH. This study can provide a reference for the application of the ozone MNBs system in the field of benzene-contaminated groundwater remediation.

Keywords: Green and sustainable in-situ remediation technology; Ozone micro-nano-bubble (OMNB); Chemical oxidation; Hydroxyl radical; Benzene

1 INTRODUCTION

Groundwater has been polluted seriously due to industrial and human activities (Kurwadkar et al., 2020). Benzene, a common volatile organic compound (VOC) in organics-contaminated groundwater, is easily volatile and harmful to public health and the environment (Sun et al., 2020). Exposure to benzene can cause a variety of health problems such as sperm abnormalities, cardiovascular disease, respiratory dysfunction, asthma, and sensitization to common antigens (Bolden et al., 2015). Therefore, it is important to study the degradation of benzene in organic-contaminated groundwater.

Currently, researchers are using various methods to degrade benzene, such as bio-remediation, advanced oxidation processes (AOPs), permeable reactive barrier (PRB), and hydrodynamic cavitation (Ma et al., 2018). However, these traditional remediation methods have limitations in terms of high costs, time efficiency, and secondary contamination. It is important to develop green and sustainable in-situ remediation techniques.

Ozone micro-nano-bubble (OMNB) technology has great potential for in situ remediation of organically contaminated groundwater due to its high oxidant transfer efficiency, high oxidation capacity, and low secondary contamination (Li et al., 2015). Some studies have used ozone MNBs to treat pollutants such as methyl orange and trichloroethylene with great results (Hu and Xia, 2018). However, ozone MNBs have not been systematically investigated in the treatment of benzene. The effects of reactant addition and groundwater matrix during ozone MNBs treatment of benzene are also not clear.

In this study, benzene-containing groundwater is treated with OMNBs in combination with hydrogen peroxide. The effects of ozone and H_2O_2 dosage, CO_3^{2-}, fulvic acid and Cl^- concentrations, and pH on the treatment of benzene-containing water are investigated through experimental studies.

2 MATERIALS AND METHODS

2.1 Reagents

Sodium chloride (≥99.9%), sodium hydroxide (≥96.0%) are obtained from Greagent (Shanghai, China). Fulvic acid (≥90.0%) is purchased from Klamar (Shanghai, China). Benzene (≥99.0%) is supplied by Meryer (Shanghai, China). Sodium carbonate (≥99.9%) is obtained from Macklin (Shanghai, China). Sulphuric acid (AR) is purchased from Tongguang (Beijing, China). Hydrogen peroxide (AR) is obtained from Xiandaidongfang (Beijing, China). Deionized water is generated by a Direct-Q® system (Millipore Corporation, France).

2.2 Production of ozone MNBs

Ozone MNBs water is generated by an MNBs generation apparatus (MBG10-A, Beijing Zhongnongtianlu Company, China). Ozone is produced by an air-source ozone generator (CFG-20Y, Beijing Bonayuan Company, China) at a concentration of 100mg/L. 10 L of deionized water with a flow rate of 0.5 L/min of ozone gas is injected into the MNBs generator. After generating for 20 min, ozone MNBs water with a dissolved ozone concentration of approximately 60 mg/L is obtained. The ozone MNBs water is then stored at 25 °C in a container ready for use.

2.3 Micro-nano-bubbles characteristics measurement

The size and concentration of ozone MNBs are the main factors affecting the mass transfer efficiency. The size distribution and the number of the ozone MNBs are measured by a nanoparticle tracking analyzer (Nano Sight LM-10, Malvern Instruments Ltd., Malvern, Worcestershire, UK). The size distribution of ozone MNBs generated in deionized water for 30 min is shown in Figure 1. The diameters of the ozone MNBs mainly ranged from 15 nm to 525 nm, and the mean diameter is 176±5 nm. The concentration of ozone MNBs is 3×10^7 bubbles per mL.

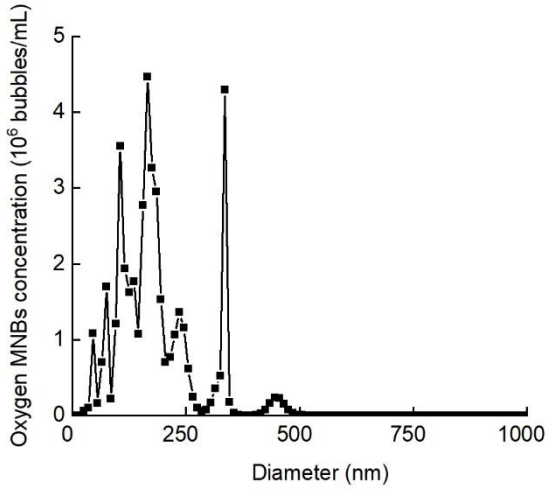


Figure 1. *Ozone MNBs size distribution*

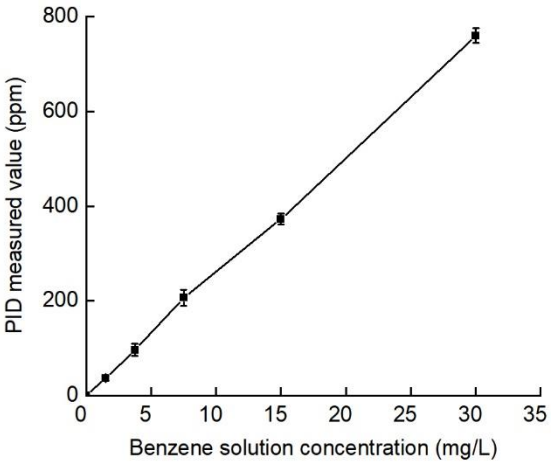


Figure 2. *PID measured value versus benzene solution concentration*

Zeta potential affects the stability of MNBs. The zeta potential of ozone MNBs is measured using the electrophoretic light scattering method (ELS) with a zeta potential analyzer (NanoBrook 90Plus Zeta, Brookhaven Instruments, USA). The zeta potential of ozone MNBs is -23.02 mV.

2.4 Benzene oxidation experiment

When the benzene oxidation experiment by ozone MNBs is carried out, 30 mL benzene solution with a temperature of 25 °C and a rotator is added to a conical flask of 200 mL before the addition of ozone MNBs, and the conical flask is placed on a magnetic stirrer with the rotating speed of 500 rpm. The concentration of benzene is selected as 30mg/L concerning the concentration of contaminants in groundwater at the actual site of a remediation project.

A certain volume of ozone MNBs and deionized water is added to the conical flask, ensuring that the total volume reaches 120 mL. Then the flask is quickly sealed. After the reaction, the reaction system continues to be kept sealed for 6h to allow the concentration of contaminants in the gas and liquid phases to reach equilibrium. Meanwhile, a blank sample is prepared, 30 mL of benzene solution and 90 mL of deionized water are added into the conical flask, and the same magnetic stirrer is used to stir and equilibrate for 6h. The glass stopper of the conical flask is then opened and the concentration of benzene in the air is determined by PID. The degradation rate of benzene is determined from the concentration in the samples after the reaction and in the blank sample.

The test on the effect of hydrogen peroxide promotion is carried out by adding a certain amount of hydrogen peroxide to adjust the molar ratio of hydrogen peroxide to ozone in the solution. The effect of chloride ions, carbonate, and dissolved organic matter on the treatment of benzene with ozone MNBs is investigated by adding a certain amount of sodium chloride, sodium carbonate, and fulvic acid to the solution respectively. The concentration gradients used in the tests are 0, 50, 100, 200, 300, 500 and 1000 mg/L. The influence of pH on the degradation of benzene by ozone MNBs is investigated by adding H_2SO_4 or NaOH solution to adjust pH between 3 and 11. The mixed solution is stirred evenly by the magnetic stirrer before the oxidant experiment. The above experiments are all repeated.

2.5 Analytical methods

The ozone concentration of ozone MNBs water is measured using an ozone concentration sensor (DOZ5000, Clean instruments co, China). Benzene concentration in the gas phase is determined by PID (GX-6000, Riken kenki co, Japan). More specifically, a solution of 0, 1.5, 3.75, 7.5, 15, and 30 mg/L of benzene is prepared and 30 mL is added to a conical flask with a volume of 200 mL. 90mL of deionized water is then added to bring the total volume to 120mL. A magnetic rotor is used to stir the solution at 500 r/min for 6 h at 25°C to equilibrate the benzene in the gas and liquid phases of the conical flask. After equilibrium is reached, the concentration of benzene in the gas phase of the conical flask is determined using PID. As shown in Figure 2, The relationship curve between the liquid phase concentration of benzene and the gas phase concentration measured by PID is plotted and fitted using a linear equation with a fit of more than 0.99. During the experiment, the concentration of benzene in the gas phase at the end of the reaction and after equilibrium is reached could be determined using PID and then the concentration of benzene remaining in the solution is determined from the fitted equation. The difference between the benzene concentrations measured using this method and those measured using gas chromatography do not exceed 5% and the method is therefore considered to be feasible.

3 RESULTS AND DISCUSSION

3.1 The kinetics of ozone self-decomposition

The previous study has shown that the decrease of dissolved ozone obeyed the first-order reaction model (Hu and Xia, 2018). The difference of self-decomposition rate between ozone MNBs is as shown follows:

$$\frac{\partial C_{\mathrm{oz}}}{\partial t} = -k_1 C_{\mathrm{oz}} \tag{1}$$

where C_{oz} is the dissolved ozone concentration (mg/L) and k_1 is the first-order decomposition coefficient (s^{-1}).

The equation can be integrated to obtain the following equation:

$$ln\frac{C_t}{C_0} = -k_1 t \quad (2)$$

where C_t is the dissolved ozone concentration at time t (mg/L), and C_0 is the initial dissolved ozone concentration (mg/L).

The ozone self-decomposition curve of ozone MNBs is shown in Figure 3. The k_1 value for ozone MNBs is 0.0432 min^{-1}. The previous study (Hu and Xia, 2018) has shown that the k_1 of ozone macro-bubbles of water is 1.428 min^{-1}. The half-life of dissolved ozone in MNBs is increased by a factor of 33 compared to macro bubbles. This suggests that the MNBs significantly delay the autolysis of ozone, allowing a constant supply of ozone to the reaction system. At the same time, the very high mass transfer efficiency of the MNBs results in a much higher concentration of dissolved ozone in the solution compared to macro bubbles. These two aspects allow more ozone to react with pollutants rather than break them down, increasing the utilization of ozone.

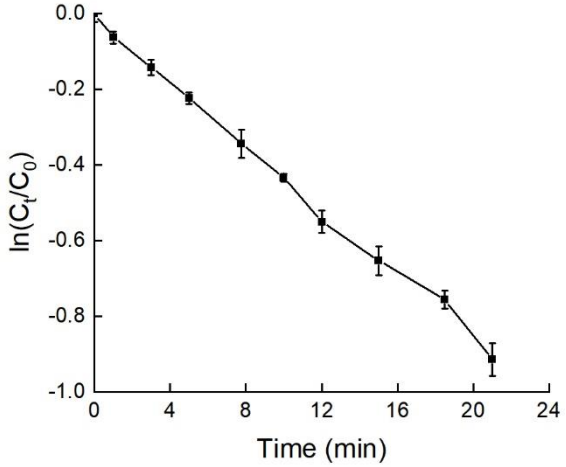


Figure 2. *Ozone self-decomposition curve in ozone MNBs*

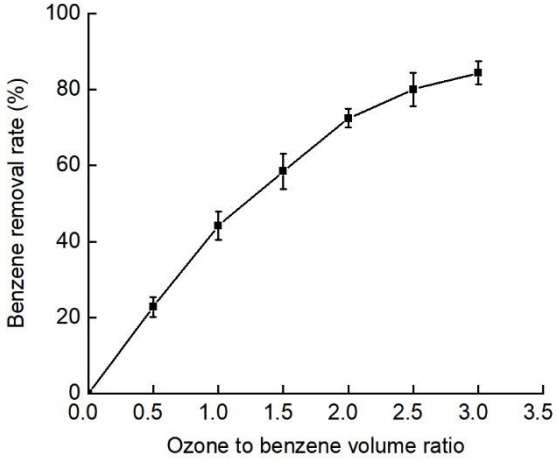


Figure 4. *Variation of benzene removal rate with ozone MNBs to benzene volume ratio*

3.2 The oxidation of benzene by ozone MNBs

The amount of ozone added is an important indicator in the process of benzene treatment by ozone MNBs, which affects the benzene removal rate and the ozone utilization rate. Different doses of ozone MNBs water are used to react with benzene to study the effect of ozone addition on benzene degradation.

The benzene removal rate varies with the volume ratio of ozone MNBs water to benzene as shown in Figure 4. The benzene removal rate is 44.17% when the volume ratio of ozone MNBs water to benzene is 1 and 84.42% when the volume ratio of MNBs water to benzene is 3. It can be seen that as the volume of ozone to benzene ratio increases with the addition of ozone MNBs water to the reaction system, the benzene removal rate also increases. In addition to the benzene removal rate, the amount of ozone required to remove a unit mass of benzene is also worth studying which can be calculated from the benzene removal rate. As the volume ratio of ozone MNBs water to benzene increases from 1 to 3, the ozone mass required to remove 1 mg of benzene gradually increases from 4.39 mg to 7.11 mg. The oxidation of pollutants by ozone mainly consists of direct and indirect oxidation, where ozone molecules can oxidize and degrade pollutants directly or by generating hydroxyl radicals(Yang et al., 2022). As the amount of ozone MNBs water added increases, the ozone utilization gradually decreases, probably due to the excess ozone in the solution not being able to make sufficient contact with the benzene, resulting in a higher percentage of autolysis of the ozone. In addition, excess ozone can cause the generated free radicals to react with each other and produce substances with less oxidizing power. This result also indicates that the ratio of ozone MNBs water to pollutant addition needs to be regulated to achieve better removal and higher ozone utilization during the actual pollutant degradation process.

In this study, we mainly focus on the degradation effect of ozone MNBs on benzene without investigating the oxidation product profile of the reaction of ozone MNBs with benzene. However, we can still make some speculations about the degradation products of benzene treated with ozone MNBs. Compare with

traditional ozone technology, ozone MNBs is able to generate more hydroxyl radicals with high oxidation capacity, which can degrade pollutants into smaller molecules and even into carbon dioxide and water. Therefore, we believe that the molecular weight of the ozonation products may be smaller and the degradation of pollutants more complete with the ozone micro-nano-bubble technology compared to the conventional ozone technology.

3.3 Influence of H_2O_2

Hydrogen peroxide, a commonly used in situ chemical oxidation remediation reagent, is also often used in conjunction with ozone to promote the production of hydroxyl radicals to enhance the removal of pollutants (Hu and Xia, 2018; Tizaoui et al., 2007). Pre-experiment with benzene treated with hydrogen peroxide alone is first carried out and shows little reduction in benzene concentration, indicating that benzene can't be degraded by hydrogen peroxide. Then, the facilitation effect of hydrogen peroxide in the treatment of benzene by ozone MNBs is investigated by adding different amounts of hydrogen peroxide to the reaction system.

The benzene removal rate varies with the molar ratio of hydrogen peroxide to ozone as shown in Figure 5. The result of the experimental treatment of benzene with hydrogen peroxide-enhanced ozone MNBs is consistent with the existing studies. As can be seen from Figure 5, hydrogen peroxide has a significant contribution to the treatment of benzene with ozone MNBs when the addition level is small. The best promotion is achieved when the molar ratio of hydrogen peroxide to ozone is 0.15 and when the benzene removal rate reaches 95.58%. However, as the amount of hydrogen peroxide added continues to increase, its facilitation effect on the ozone MNBs treatment of benzene gradually diminishes. When the molar ratio of hydrogen peroxide to ozone is increased from 0.15 to 0.75, the benzene removal rate is reduced from 95.58% to 87.3%. This transition may be related to the reaction between hydrogen peroxide and hydroxyl radicals. Increasing the amount of hydrogen peroxide would change its role from that of an initiator of hydroxyl radical generation to that of an inhibitor of ozone breakdown through free radical reactions. The scavenging effect of hydrogen peroxide on hydroxyl radicals makes it possible to have an optimal dosage, beyond which there is a decrease in the promotion effect (Tizaoui et al., 2007). Therefore, at higher hydrogen peroxide additions, the excess hydrogen peroxide reacts with the hydroxyl radicals, resulting in a lower benzene removal rate.

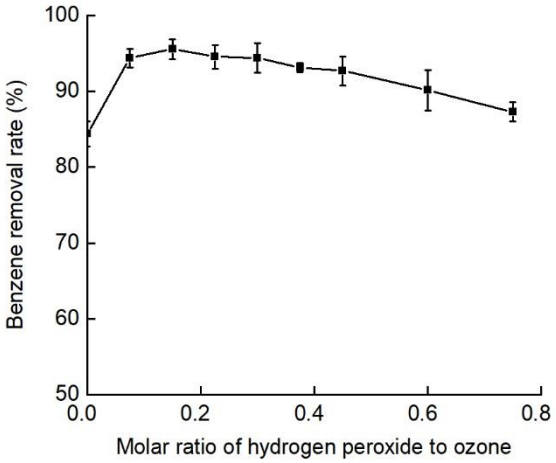


Figure 3. *Variation of benzene removal rate with the molar ratio of hydrogen peroxide to ozone*

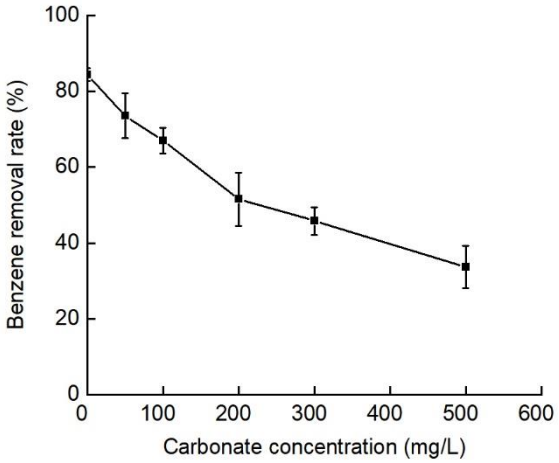


Figure 6. *Variation of benzene removal rate with carbonate concentration*

3.4 Influence of inorganic ions

The ions contained in the groundwater matrix can have an impact on the oxidative degradation process of contaminants. In this study, the influence of common ions on the degradation of benzene by ozone MNBs is investigated, including Cl^- and CO_3^{2-}, which are representative of groundwater.

The effect of carbonate on the treatment of benzene by ozone MNBs is shown in Figure 6. It can be seen that the removal of benzene by ozone MNBs decreases as the carbonate concentration in the solution continues to increase. When the carbonate concentration reaches 500 mg/L, the benzene removal rate decreases from 84.42% to 33.66%. This suggests that carbonate has a significant inhibitory

effect on ozone MNBs treatment of benzene. This inhibition is because carbonate is a quencher of hydroxyl radicals (Khuntia et al., 2013). The carbonate and bicarbonate radicals can react with hydroxyl radicals, converting the strongly oxidizing hydroxyl radicals into the less oxidizing carbonate and bicarbonate radicals (Asghar et al., 2022; Pan et al., 2022). Therefore, as the carbonate concentration in the solution increases, the hydroxyl radicals are consumed, leading to a reduction in the removal of benzene.

The effect of chloride ion concentration on the treatment of benzene with ozone MNBs is shown in Figure 7. In complete contrast to the case of carbonate, the presence of chloride ions in the solution contributes to the removal of benzene. The benzene removal rate increases from 84.42% to approximately 90% in the presence of chloride ions. But continued increases in chloride ion concentration don't result in a further increase in benzene removal. It is commonly believed that the chloride ions in the solution deplete the reactive oxygen species such as ozone and hydroxyl radicals in the solution, which makes the removal rate of pollutants decrease. But this is not consistent with the result of this experiment. In this experiment, the facilitation of benzene removal by chloride ions may be due to the more selective radicals generated by the reaction of hydroxyl radicals with chloride ions, which can more effectively eliminate electron-rich micro-pollutants from saline water (Asghar et al., 2022; Zhang and Parker, 2018). However, the effect of chloride ions on the treatment of benzene with ozone MNBs needs to be investigated in more depth to determine if more toxic by-products are produced in the reaction system in the presence of chloride ions. The experimental results on the effects of carbonate and chloride ions on benzene treatment by ozone MNBs indicate that the groundwater matrix has a significant influence on the contaminant degradation process and needs to be considered when carrying out practical projects.

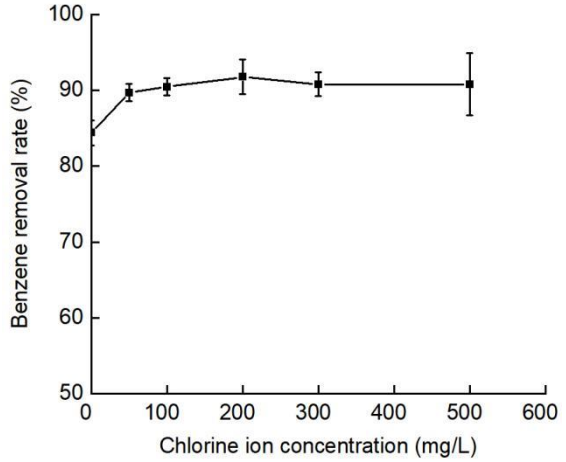


Figure 4. *Variation of benzene removal rate with chlorine ion concentration*

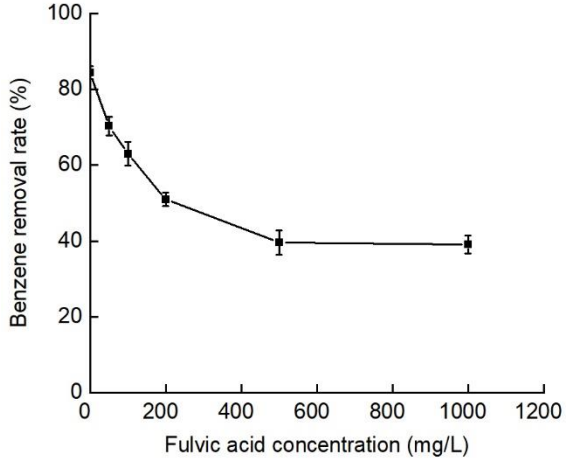


Figure 8. *Variation of benzene removal rate with fulvic acid concentration*

3.5 The influence of dissolved organic matter (DOM)

The prevalence of DOM in groundwater can interfere with the oxidation process of contaminants in groundwater. The effect of DOM on the treatment of benzene by ozone MNBs is investigated using fulvic acid as a proxy for dissolved organic matter, as shown in Figure 8. We can find that the effect of fulvic acid is significant, and it has a strong inhibitory effect on ozone MNBs treatment of benzene even when its concentration is low. At a fulvic acid concentration of 200 mg/L, the benzene removal rate is reduced from 84.42% to 50.99%. When the fulvic acid concentration is increased to 500mg/L, the benzene removal rate is reduced to 39.59%, and the benzene removal rate does not decrease when the fulvic acid concentration is increased.

The specific mechanism of DOM inhibition of benzene treatment by ozone MNBs is the depletion of dissolved ozone and hydroxyl radicals in the water (Asghar et al., 2022; Yang et al., 2022). Dissolved organic matter may be more susceptible to degradation by ozone MNBs than benzene. When ozone MNBs are added to contaminated groundwater, they may react preferentially with DOM rather than benzene, which can result in additional ozone depletion and reduced contaminant removal. Although some studies have pointed out that DOM can also react with ozone to generate hydroxyl radicals, this process doesn't dominate in this experiment. Therefore, when treating benzene with ozone MNBs, it is

necessary to determine the concentration of DOM in the water. Reasonable ways need to be used to reduce the effect of DOM and to prevent the oxidant from being depleted rather than used to treat the contaminant.

3.6 The influence of pH

The effect of pH on the treatment of benzene with ozone MNBs is shown in Figure 9. We can find that the ozone MNBs are most effective in treating benzene under weakly acidic conditions. As the pH continues to increase, the degradation rate of benzene continues to decrease. At pH 5, benzene removal reaches 91.26%, while at pH 11, benzene removal decreased to 73.57%. Studies have shown that under acidic conditions, ozone decomposes slowly, while under alkaline conditions, the ozone molecule becomes unstable, resulting in a rapid decrease in the concentration of ozone in the solution. In alkaline environments, ozone molecules readily decompose to produce very short-lived hydroxyl radicals, which will rapidly die out if they fail to come into contact with pollutants in time. When ozone MNBs are added to contaminated groundwater, they may react preferentially with DOM rather than benzene, which can result in additional ozone depletion and reduced contaminant removal. In addition, the strongly oxidizing radicals in solution under alkaline conditions may react with each other to form substances with less oxidation capacity. It is also shown that as the pH increase, ozone exposure decreases significantly while hydroxyl radical exposure remains unchanged, which led to a decrease in the degradation rate of pollutants under alkaline conditions (Elovitz et al., 2000). The pH of groundwater is generally neutral, which facilitates the degradation of contaminants.

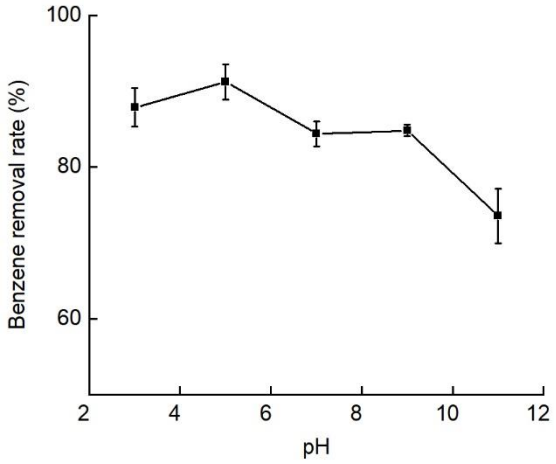


Figure 5. *Variation of benzene removal rate with pH*

Experiments on the effects of inorganic ions, dissolved organic matter, and pH in groundwater on the treatment of benzene with ozone MNBs have shown that environmental factors in groundwater have significant effects on the degradation of contaminants. However, in actual remediation projects, the coexistence of target contaminants with organic and inorganic substances in groundwater is very common and complex, and the applied ozone MNBs can react with these groundwater matrices, resulting in the contaminant removal effect being affected. Therefore, it is necessary to perform some pre-treatment of groundwater before it enters the ozone MNB treatment unit. In actual groundwater remediation projects, ozone micro-nano-bubble technology can be used in combination with other technologies such as flocculation, activated carbon filtration, or adding some chemicals to the water to remove some of the inorganic and organic compounds. By these means, the effect of factors other than the target contaminants in groundwater can be reduced and the effectiveness of ozone micro-nano-bubble technology in treating contaminants in groundwater can be enhanced.

4 CONCLUSIONS

In this paper, the degradation of benzene in water by ozone MNBs is investigated through batch experiments. The study shows that ozone MNBs technology combined with hydrogen peroxide is an efficient method to treat benzene-contaminated groundwater. The main conclusions are as follows:

The half-life of ozone in MNBs is 33 times longer than that in macro bubbles and MNBs extend the lifetime of ozone and improve ozone utilization. As the ozone dose increases, the degradation rate of benzene gradually increases. When the volume ratio of ozone to benzene is 3, the benzene degradation rate is 84.42% and the amount of ozone required to remove 1 mg of benzene is 7.11 mg.

Hydrogen peroxide is proven to enhance the removal efficiency of benzene when combined with ozone MNBs as compared to using ozone MNBs or hydrogen peroxide alone. The optimal hydrogen peroxide to ozone molar ratio of 0.15 is a limiting ratio above which a decline in the benzene removal rate.

Carbonate and fulvic acid in the water will inhibit the chemical reaction system by reacting with ozone and hydroxyl radicals. When the concentration of carbonate and fulvic acid is increased to 500 mg/L, the benzene removal rate decreases from 84.42% to 33.66% and 39.59% respectively. The benzene treatment is facilitated by chloride ions.

The radicals of chlorine formed by the reaction of hydroxyl radicals with chloride ions in solution may have a better treatment effect on benzene When the chloride ion concentration is increased to 500 mg/L, the benzene removal rate increases to 90%.

The best removal of benzene is achieved when the solution is weakly acidic, with a gradual decrease in removal as the pH increases. This may be due to the instability of ozone under alkaline conditions, while the generated hydroxyl radicals are too short-lived and may not adequately contact the contaminants.

This study may provide a reference for the application of ozone MNB technology in groundwater remediation contaminated with benzene.

5 ACKNOWLEDGEMENTS

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