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Oxidative degradation technology of synthetic water pollutants containing sulfamethoxazole in urban suburbs based on section water quality response

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ABSTRACT

In order to solve the problem of drug and pesticide pollution in the river water in the suburbs of the city. The degradation experiments of sulfamethoxazole (SMX) and atrazine (ATZ) were designed. The effect of degrading strain was tested by nano gold chip. In the degradation experiment of sulfamethoxazole, five kinds of SMX degrading strains were screened out. The concentration of SMX before and after the degradation of SMX degrading strains was determined to determine the degradation rate of antibiotics by different strains. The results showed that HA-J1 was the most efficient sulfonamides biodegradable strain, and the degradation rate was up to 65%. The results show that 88% of ATZ can be removed in 10 s under the condition of ferrate concentration of 100 μmol/L and ATZ concentration of 10 μmol/L. The above experimental results show that the technology in this paper is helpful to the relevant treatment of actual water pollution.

Keywords: Atrazine; Nanogold gene chip; Water pollutants; Antibiotics; Ferrate activates sulfites

1. Introduction

At present, drug organic pollutants represented by antibiotics and pesticide pollution represented by ATZ have great potential impact on human health and ecosystem in reclaimed water [1]. Mariculture activities and river input may lead to the pollution of trace pollutants (such as antibiotics and pesticides) in coastal seawater. Therefore, it is of great significance to investigate the occurrence and risk of organic micro pollutants in coastal waters. In this study, 13 antibiotics and 15 pesticides were screened in the coastal waters of Liaodong Peninsula, China. Of these targets, 13 were antibiotic detected at concentrations as high as 64.8 ng/L. The concentration is below 100 mg/L, indicating that the antibacterial effect is weak. It was found that cimazine, ATZ and triazolol were the main pesticides, and the detection frequency was 100%. Sulfamethoxazole was the main antibiotic, and the detection frequency was 62.5%. The total amount of pollutants in the Bohai Sea is generally higher than that in the Yellow Sea, and shows a decreasing trend to the sea. Through principal component analysis, land input and mariculture are divided into the main pollution sources [2]. Bacteriophages (phages) are viruses that infect bacteria. The "Predator–Prey" interactions are recognized as a potentially effective way to treat infections. Bacteriophages and phage derived proteins, especially enzymes, have been intensively studied to become alternative or supportive antibiotics used alone or in combination with standard antibiotic regimens in the future. The advantages and limitations of the two drugs in specificity, mode of action, structural problems, drug resistance development, pharmacokinetics, product preparation and interaction with the immune system were discussed. Finally, the current regulations for the application of bacteriophage based products are described [3].

The prepared nanocomposites were characterized by various techniques (such as SEM-EDS (scanning electron microscope + energy dispersive spectrometer), FTIR (Fourier transform infrared spectroscopy), XRD (X-ray diffraction), BET (Brunner–Emmet–Teller), XPS (X-ray photoelectron

spectroscopy) to clarify the successful loading of HMO and analyze the subsequent adsorption mechanism. The effects of adsorbent dosage, initial concentration of adsorbate, reaction time, solution pH and temperature on the reaction were investigated. The adsorption kinetics of removal showed that the equilibrium was reached within 12 h, and the removal rates were 91.9% and 99.5%, respectively. The corresponding adsorption data accorded with the second-order kinetic model [4]. Corrigendum to "One-pot fabrication of rod-like magnesium silicate and its adsorption for Cd^{2+}" [5]. Membrane crystallization for salts recovery from brine-an experimental and theoretical analysis [6]. To study the effect of substituents on the genotoxicity of benzophenone filters [7].

With the development of environmental analysis technology and people's increasing concern for their own health, the "emerging pollutants" in water environment have gradually become the focus of environmental chemistry research. The biological activity and toxicity of water pollution have attracted much attention. In water and soil environmental media, the frequency of detection is the highest in surface water, especially common antibiotics and atrazine.

In recent years, with the wide use of antibiotics in clinics, a large number of antibiotic residues in emissions to water all over the world, the main water antibiotics can produce the selection pressure of microbial resistance to the environment and the selectivity of resistant pathogens, causing serious pollution of surface water and groundwater, in the human body and aquatic life soon appeared resistance, super drug-resistant bacteria, to human health and a serious threat [8]. How to scientifically and effectively remove antibiotic residues in water is the focus of close attention of researchers at home and abroad. In addition, ATZ can be frequently detected in surface water and groundwater in many countries and regions, which is one of the highest detection rates of pesticides in groundwater and surface water. ATZ has the characteristics of stable molecular structure and difficult degradation, and the traditional biological treatment technology is not ideal for ATZ treatment. In addition, the adsorption method and oxidation method also have problems such as low adsorption efficiency, high treatment cost, long degradation time and unsatisfactory treatment effect when treating ATZ. In order to solve the problems of antibiotic residues and pesticide pollution in the river in the suburbs of the city. Therefore, it is of great practical significance to study an efficient and environmental protection method for water treatment of antibiotics and ATZ. Moreover, the treatment effect of water pollution was optimized.

2. Oxidative degradation of river water pollutants in urban suburbs

2.1. Nano gold gene chip technology

According to the research, compared with the traditional biological treatment technology, the Nano gold tablet has better labeling performance and higher detection sensitivity, and the state changes of the tested strains can be obtained directly. Nano gold refers to tiny gold particles, soluble in water, strong binding, can be detected without affecting the activity of the tested strains, and the results are obvious, easy to experimental analysis. Therefore, in this study, we used the Nano gold gene chip technology for strain degradation, in order to obtain accurate experimental values.

2.2. Degradation of sulfamethoxazole antibiotics

2.2.1. Research methods

At first, the sulfamethoxazole (SMX) degrading strain was isolated from the sludge of municipal sewage treatment plants. The degradation rates of different strains against SMX were studied, and the highly degradable strains were determined. According to the experiment, further verify whether SMX can be used as the only carbon source, and determine the strain through sequencing.

It is known that the DNA sequencing result of the strain is compared with the authoritative literature, there is no change in the composition or sequence of base pairs in the structure of the gene and no gene mutation is found. The treatment process of aerobic biological treatment is that microorganisms use the organic pollutants in water as low substances for aerobic metabolism, release energy step by step through a series of biochemical reactions, and finally stabilize with low-energy inorganic substances to meet the requirements of harmlessness, For return to the natural environment or further treatment. The MLVSS (mixed liquid volatile suspended solids) concentration is 0.75 and the sludge residence time is 12–24 h.

In this paper, a part of the residual sludge is treated as a sludge sample in the sludge thickener of a sewage treatment plant for household cleaning products in the suburbs of a city.

2.2.2. Problems to be solved

Purification, culture and screening of sulfamethoxazole efficient degrading strain were considered in this step. Moreover, the degradation rate of sulfamethoxazole degrading strain was detected by gold nanoparticle microarray [9]. Finally, the degradation characteristics of sulfamethoxazole were evaluated.

2.2.3. Degradation experiment of sulfonamides antibiotics

The data of this paper are taken from the concentrated sewage of a river washing and nursing product sewage treatment plant in the suburb of a city. Repeat the experiment 3 times to ensure the accuracy of the experiment. After being diluted to different concentrations, SMX-resistant bacteria are screened out by coating and separation on the LB plate with SMX added. The concentration of SMX was determined by HPLC, and the test time was 1–2 h. Then select the single strain with better growth, realize the crossing separation, until the purification. Single strains were cultured in medium supplemented with inorganic salt, the only carbon source of SMX, the culture medium includes peptone, meat extract, inorganic salts, growth factors, beef extract, sugars, eggs, animal serum and blood. The

concentration of inoculum is 0.5 MCU, and the concentration of degradation compound is 1~200 mg/L, to screen out the strains capable of decomposing SMX. The degradation rate of SMX strain per unit time was determined by gold nanoparticle microarray. At the same time, several strains with high degradation efficiency were selected and morphological observation was carried out by microbial staining technique. At the same time, experiments were carried out with different degradation rates of SMX concentrations to test the degradation kinetics of the strains and whether they complied with the Monod equation. The strain was determined by sequencing and comparison.

1 g of the mud sample was mixed in 9 mL sterile water, and then 1 mL of the bacterial liquid was diluted by 10 times. According to this method, diluent of bacterial liquid was obtained by 101–107 times. At the same time, take 100 μL of diluent 103–107, evenly coat it on sulfonamides medium LB plate through coater, and put it into an incubator with constant temperature and electric heating, the temperature in the incubator is 30°C.

Sulfa LB solid culture medium formula for selected: peptone 10 g, 12 g AGAR powder, yeast extract, 5 g, 800 mL of distilled water, sodium chloride l0 g, at the same time adjusting the pH of 7, the capacity to 1 L for high pressure sterilization (121°C, 20 min), add the chef to SMX solution concentration is 100 μg/mL, complete shake well and pour plate operation, at 4°C storage for later use.

2.2.4. Screening SMX resistant bacteria

For 24 h after the training of the appropriate dilution degrees sulfa, tablet, and medium forms of single colony find appearance, scrape by inoculating loop, respectively in sulfa, selection of medium plate, and separate line on the tablet, the 3 copies of each strain parallel placed thermostatic cultivation in 24 h. Repeat the process 2–3 times until you are sure to screen out about 10 strains of SMX-resistant bacteria.

2.2.5. Screening of SMX degrading strains

Single strain 24 h after training, to scrape by inoculating loop sulfa, and the single strain of medium plate, in 250 mL of inorganic salt liquid medium, joined with the SMX solution, until the final concentration of mu 100 μg/mL, shaking culture under the 30°C after 48 h, the growth of better strains, was conducted in the sulfa selection of medium plate, each kind of strain 2 parallel stored for later use. If different forms of bacteria or single strains did not form in the sulfonamides selection medium plate in the previous step, then the lines were drawn in the sulfonamides selection medium plate for separation [10,11].

Among them, the medium formula of inorganic salt liquid is:

A. Medium/L: KH_2PO_4 1 g, $FeSO_4$ 0.01 g, $K_2HPO_4{\cdot}3H_2O$ 3 g, $CaCl_2$ 0.005 g, $MgSO_4{\cdot}7H_2O$ 0.3 g, $(NH4)_2SO_4$ 0.5 g (The content of nitrogen element is 106mg), NaCl 1 g, pH 7.2–7.3, 1 mL of trace elements;

B. Trace element solution/L: $Na_2Mo_4{\cdot}H_2O$ 6.7 g, $CuSO_4{\cdot}5H_2O$ 2 g, $ZnSO_4{\cdot}5H_2O$ 28 g, $MnSO_4{\cdot}5H_2O$ 4 g, $CoSO_4{\cdot}7H_2O$ 4.7 g, H_3BO_4 4 g, pH 7.2.

According to the above methods, five representative strains were obtained as follows: HA-J1, HA-J4, HA-J5, HA-J7, HA-J9.

2.3. Oxidative degradation of ATZ in water by ferrate-sulfite system in response to sectional water quality

2.3.1. Determining factor of water quality response uniformity of section

Outlet of close area pollutant concentration distribution in the traditional research, define the section on the difference between maximum concentration and the minimum concentration of not more than 5%, thinks that the section on the applicable reached the mixing, called this section fully mixing section [12,13]. the discharge outlet downstream reach fully mixing section before the river called the mixing process and mixing process length and suburbs only river hydrological eigenvalue about itself [14]. It is the ideal situation without considering the suburbs outside the experience formula of the factors affecting river hydrological eigenvalue [15,16]. The influence of the discharge concentration of the upstream outfall and the background concentration of the water quality of the suburban river on the water quality distribution of the mixed section is not considered. In view of the traditional research model of this case is not sex, this article puts forward the concept of "section water quality uniformity", as a criterion to determine whether water quality monitoring section of the uniform: define any channel direction perpendicular to the suburb in the damage zone section, which is a water quality factor, the minimum density and the ratio of the maximum density, the ratio is a water quality factor in the section of the cross section in water evenness. When the water quality uniformity of cross section $\eta \geq 95\%$, it is considered that the concentration distribution of the water quality factor is uniform on the cross section [17], meeting the requirements of automatic monitoring of water intake.

Section water quality uniformity:

$$\chi = \frac{C_{\min}}{C_{\max}} \times 100\% \tag{1}$$

where χ is the water quality uniformity of section; $C_{\min}$ is the minimum concentration value of water quality factor at cross-section, mg/L; $C_{\max}$ is the maximum concentration value of water quality factor at cross-section, mg/L. In the calculation of section concentration, to simplify the model, the section is divided into 1 m units for calculation. Based on the calculated results of water quality uniformity in the above sections, the oxidation degradation experiment of ferrate and sulfite system was designed.

2.3.2. Experimental design of oxidation degradation of ferrate-sulfite system

The ferrate crystal used in this experiment is a self-made product. First, ferrate solution was prepared by electrolysis method, and then the ferrate crystal (purity ≥ 92%) was prepared after crystallization, dehydration, desalination and vacuum drying [18]. Atrazine (ATZ, purity ≥ 97%)

was purchased from Shanghai Yuanye Biotechnology Co., Ltd. Methanol (chromatographic purity) was purchased from Shanghai Sigma-Aldrich Chemicals Co., Ltd. Other reagents, including sodium sulfite, sulfuric acid, phosphoric acid, sodium hydroxide, dipotassium hydrogen phosphate, sodium tetraborate, ethanol, tert-butanol, acetone, hydroxylamine hydrochloride and so on, are all analytical pure purchased from Sinopharm Chemical Reagents Co., Ltd. [19,20].

Determination of ATZ in water samples by nanogold gene chip, Thermofisher scientific symmetry C18 column (4.6 mm × 150 mm × 5 μm); The mobile phase was methanol and water, and the volume ratio of methanol: water is 7:3. The flow rate of mobile phase was 0.8 mL/min, the column temperature was 30°C, the detection wavelength was 223 nm, and the injection volume was 20 μL. The concentration of ferrate was determined at 510 nm by gold Nano Chip [21].

The concentration of ATZ reserve solution was 100 mol/L. The solution was prepared once a week and stored at 4°C in a dark environment. Configuration: dissolve the required amount of ATZ in 5 mL acetone, and then volume it to 1,000 mL with deionized water. Ferrate reserve solution was prepared with 5mmol/L phosphate buffer solution and 1 mmol/L borate buffer solution. Both ferrate solution and sulfite solution used in the experiment were prepared before the experiment started [22,23]. ATZ degradation experiments were carried out at room temperature, and the concentration of ATZ in the water sample used in this experiment was set at 20 mol/L. Due to the decomposition of ferrate itself will produce a large number of hydroxide ions (OH^-) to improve the alkalinity of the acceptor water. In order to stabilize the pH of the system, this experiment was all carried out in phosphate buffer solution. Before the start of the reaction, the required concentration of sulfite solution to join contains 100 mL ATZ (40 μmol/L) of the buffer salt solution, and 0.1 mol/L of phosphoric acid and 0.1 mol/L sodium hydroxide solution acidity of the solution pH, respectively to below target and add the same volume of ferrate liquid reserves (200 μmol/L), when the solution pH to response the required pH (5.0, 7.0, 9.0) reaction started. At the set time, 5 mL samples were taken out of each beaker with a pipetting gun, and immediately added into a small beaker containing 10 μL hydroxylamine hydrochloride solution (500 mmol/L) to terminate the reaction. Then, the filtered samples were filtered by a 0.45μm cellulose acetate filter membrane, and injected into a liquid chromatography vial for subsequent liquid chromatography detection [24].

3. Experiment

3.1. Degradation performance of SMX degrading strain was determined

The single strain that grew well in the inorganic salt liquid medium was regarded as SMX degrading bacteria, and the degradation ability was measured. Will be 1% inoculation quantity, in liquid medium, vaccination to 250 mL of liquid inorganic salt medium, join now with the SMX solution at the same time, until the final concentration is 100 μg/mL, shaking culture under the 30°C after 12 h, the degradation rate of sulfonamide antibiotics was obtained by using the technology of nanogold microarray to determine the SMX concentration of the strains before and after culture. Five representative strains: HA-J1, HA-J4, HA-J5, HA-J7 and HA-J9 were used to characterize and analyze the degradation rate of sulfonamides antibiotics [25,26] in urban sewage treatment plants, and lay a foundation for future research. The test results are shown in Table 1.

According to Table 1, the five strains all degraded the sulfonamides of SMX to different degrees. The HA-J1 strain had the highest physical degradation rate of 65%, followed by the other strains. Among them, the degradation rate of HA-J4 was second only to that of HA-J1, while HA-J9 had the lowest degradation rate of SMX. In the comparison of biodegradation rate, the degradation rate of HA-J1 strain was also the highest [27]. In conclusion, HA-J1 pair is the first choice for SMX degradation.

3.2. Experiments on morphological observation and degradation kinetics

According to the experiment of the previous step, HA-J1 and HA-J4, two relatively efficient SMX degradation strains, were selected to realize the observation of morphological characteristics and physiological characteristics. At the same time, the OD_{600} values before and after the liquid culture medium were measured to obtain the specific growth rate of the medium at different concentrations of SMX, and then the degradation kinetics was verified to be consistent with the Monod equation.

The linear simplified formula of Monod equation is usually used to solve whether the dynamics of strain degradation conforms to the Monod equation or not. The reciprocal solution formula of Monod equation is as follows:

$$\frac{1}{v} = \frac{1}{v_{\max}} + \frac{K_s}{v_{\max}} \cdot \left(\frac{1}{S}\right) \tag{2}$$

where v represents the specific degradation rate of SMX, and S represents the concentration of SMX. If $1/v$ is plotted against $1/S$, a straight line can be obtained, and the intercept of the vertical axis is $1/v_{\max}$ and the slope is $K_s/v_{\max}$. So we can figure out what $v_{\max}$ and K_s are.

In order to establish the kinetic equation for the degradation of SMX by the strain, the degradation rate of SMX by the experimental strain fitted with the Monod equation was solved under the optimal fixed conditions. The concentration of SMX in the medium was 100 mg/L, and the

Table 1
Physical degradation rates of five strains to sulfamethoxazole

Strain	Degradation rate (%)
HA-J9	0.15 ± 0.05
HA-J7	0.48 ± 0.06
HA-J5	0.33 ± 0.05
HA-J4	0.64 ± 0.05
HA-J1	0.65 ± 0.15

residual SMX concentration in the medium was measured every 2 d. The results are shown in Fig. 1.

In order to obtain the parameters v_{max} and K_s in the Monod equation, in the linear interval, the linear simplification method is used to plot 1/v against 1/S, that is, the reciprocal of the degradation rate of SMX and the reciprocal of the concentration of SMX, and the correlation coefficient is 0.9904. The result is shown in Fig. 2.

According to Fig. 3, the kinetic equation of SMX degradation by the strain is as follows:

$$v = \frac{v_{max} \cdot S}{K_S + S} \tag{3}$$

The actual degradation rate of the strain under different concentrations of SMX can be obtained by calculation. According to the degradation rates of different strains at different concentrations of SMX, the actual degradation rate of strains will be greater than the maximum degradation rate v_{max} of Monod equation when the concentration of SMX is relatively high. When the concentration of SMX was relatively low, the actual degradation rate of the strain was close to the result of the Monod equation, which proved that the degradation kinetics of sulfonamides SMX complied with the Monod equation. SMX can be used as the only carbon source of the degradation strain.

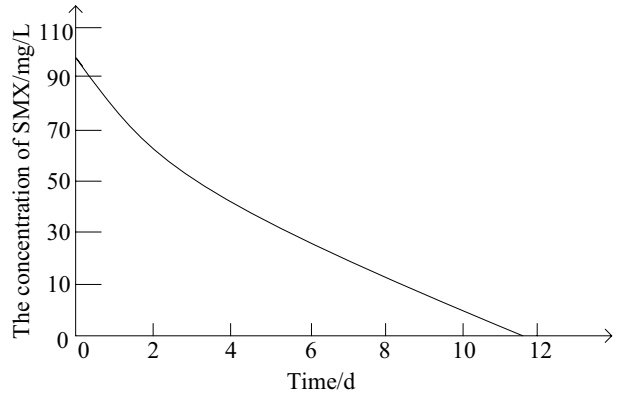

Fig. 1. Changes of SMX concentration over time.

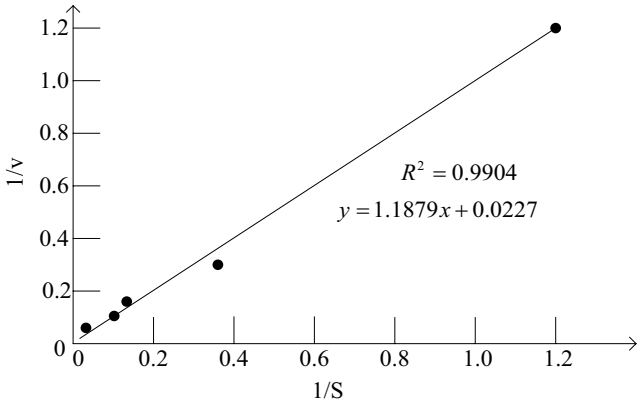

Fig. 2. Fitting curves of 1/v and 1/S.

3.3. Comparison of ATZ degradation effects at different ferrate concentrations

The presence of sulfites can only generate active radicals to degrade the target contaminant ATZ in the system after being activated by ferrate. The optimal dosage ratio of ferrate to sulfite is 1:4. Then, under the condition of fixing this dosage, the concentration of ferrate is increased for testing. The experimental results are shown in Fig. 3.

The removal rate of ATZ decreased with the increase of ferrate concentration. When the ferrate concentration was 100 μmol/L, the ATZ removal rate of the system was 45%. With the increase of ferrate concentration, the ATZ removal rate decreased to 36% and 30%.

It should also be noted that the concentration of sulfites in the system increased with the increase of ferrate concentration after the fixed dosage of gaby. One possible reason is that after the ferrate and sulfite concentration, the system of ferrate more activation of the sulfite in the system, the quantity of the activity of free radicals increases greatly, however, a high concentration of SO4 – self quenching, consumption has been greatly intensified, involved in the degradation of ATZ instead of active free radicals concentration decreased. Another possible reason is that activation of sulfite ferrate can only happen when in low concentrations of sulfite, even also improve ferrate and concentrations of sulfite, ferrate can only activate certain concentrations of sulfite, and system of excess instead of sulfite consumes the activity of free radicals, resulting in a loss of ATZ removal rate.

In order to further explore the effect of ferrate concentration on ATZ degradation in ferrate-sulfite system, the experiment was carried out by changing the sulfite concentration under different ferrate concentration. The experimental conditions were set as follows: ferrate concentration was 100 μmol/L, 200 mol/L, 400 mol/L, sulfite concentration was 10~2,000 μmol/L, ATZ concentration was 20 μmol/L, pH = 9.0.

The experimental results are shown in Fig. 4. Under different ferrate concentrations, the ATZ removal rate in the system increases first and then decreases with the increase of sulfite concentration, which is consistent with

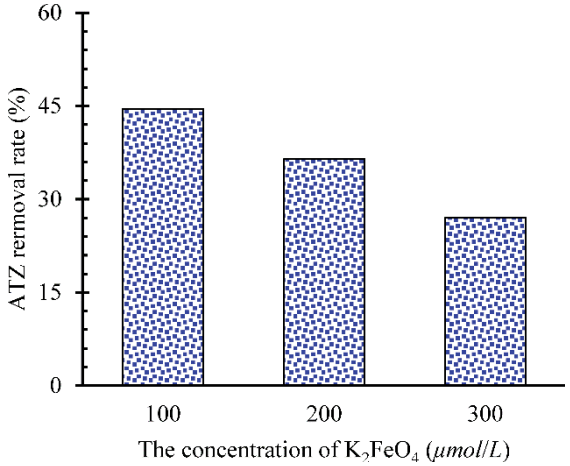

Fig. 3. ATZ removal effect of ferrate-sulfite system at different ferrate concentrations.

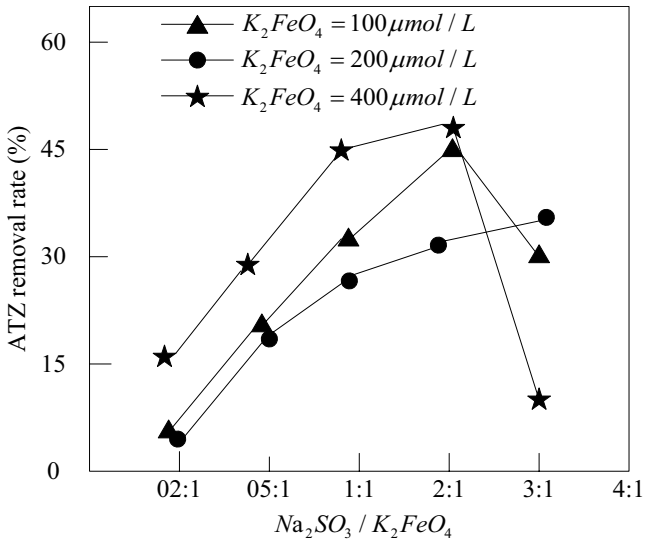

Fig. 4. Influence of the ratio of sulfite and ferrate concentrations on ATZ removal under different ferrate concentrations.

the results obtained in our previous experiment. In addition, it can also be found that with the increase of ferrate concentration, the highest ATZ removal rate that can be achieved in the system also increases. However, the optimal ratio of $[Na_2SO_3]$:$[K_2FeO_4]$ did not remain constant with the increase of ferrate concentration, but decreased accordingly. When the ferrate concentration in the system is 100 mol/L, the optimal $[Na_2SO_3]$:$[K_2FeO_4]$ = 4:1. When the ferrate concentration in the system is increased to 200 μmol/L and 400 μmol/L, the optimal dosage ratio is gradually reduced from 4:1 to 2:1 and 1:1. However, under the optimal dosage conditions, different ferrate concentrations correspond to the same sulfite concentrations, which are all 400 μmol/L. This result shows that in the ferrate-sulfite system, the concentration of sulfite in the system is closely related to the concentration of $SO_4^{\bullet-}$. The self-ego consumption between $SO_4^{\bullet-}$ shows that the concentration of $SO_4^{\bullet-}$ in the system is not the greater the better, nor can a larger concentration be reached. The concentration of ferrate and sulfite should be well controlled to make the concentration of $SO_4^{\bullet-}$ in the system reasonable, which will be conducive to the removal of the target pollutants.

4. Conclusion

In order to study the water pollution of rivers in the suburbs of cities, the degradation experiments of SMX and ATZ were carried out by using nanogold gene chip. Results confirmed that HA-J1 had an effective degradation effect on sulfonamides antibiotics in river water pollution in urban suburbs, and the degradation kinetics of sulfonamides antibiotics was proved to conform to the Monod equation, and it was also confirmed that SMX could be the only carbon source of the degradation strain. The effect of ferrate concentration and ATZ concentration on the degradation of ATZ by ferrate-sulfite system was systematically investigated in this experiment. Under the experimental conditions of ferrate concentration of 100 μmol/L and target pollutant ATZ concentration of 10 μmol/L, 88% of ATZ can be removed in less than 10 s. Furthermore, according to the achieved results, the degradation of sulfamethoxazole (SMX) and ATZ lacks consideration of its influencing factors. Control the concentration of ferrate and sulfite to make the $SO_4^{\bullet-}$ concentration in the system reasonable, which is conducive to the removal of target pollutants. In order to make the research results more reliable, it is necessary to comprehensively analyze the influencing factors of pollutant degradation, so as to further optimize the improvement effect of water resource pollution.

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