



Removal of dissolved sulfides in aqueous solution by activated sludge: mechanism and characteristics



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HIGHLIGHTS

- Dissolved sulfides removal is primarily attributed to adsorption under low DO condition in activated sludge recycling process.
- SEM/EDS, XPS, and FTIR results show that sulfides could be bonded with the primary amine group in the activated sludge.
- Sulfides adsorption isotherm fit the Langmuir model well, and kinetic fit the pseudo-second-order model well.

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ABSTRACT

Activated sludge recycling has been developed as a novel technique to directly prevent volatile sulfides emission from wastewater influents. In this study, mechanisms and characteristics of dissolved sulfides removal in aqueous solution by activated sludge were investigated. When DO content in water was 0.49 mg/L, 70% of removed dissolved sulfides were released back from the activated sludge by lowering pH to 1. The SEM/EDS result revealed that removed sulfur was fixed in activated sludge and the XPS result showed that fixed sulfur had an oxidation state of -2 . FTIR results showed that primary amine group ($R-NH_2$) could be one of the radical groups bonding sulfides. All these results verified that sulfides removal by activated sludge is primarily attributed to adsorption, rather than biodegradation, under low DO conditions in 40 min. The equilibrium isotherm data fit the Langmuir isotherm model well. The maximum adsorption capacity (q_0) ranged in 25–38 mg/g at temperatures of 10–40 °C. The adsorption kinetic data fit the pseudo-second-order model well. The amounts of adsorbed sulfides at equilibrium (q_e) were positively proportional to temperature, initial sulfides concentration and agitation speed. These results indicate that sulfides adsorption could be a chemical sorption or ion exchange process.

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1. Introduction

Odor contamination recently became a significant environmental problem in the world. Typical odorous compounds are predominantly volatile fatty acids, phenols, ammonia, volatile amines, and volatile sulfur-containing compounds [1]. Hydrogen sulfide (H_2S) is considered to be a dominant contributor of odorous compound emission for municipal wastewater treatment plants (WWTPs). Moreover, high concentrations of H_2S are highly corrosive on the sewers of WWTPs and carry toxicity for living organisms [2,3].

Several methods are in use to control H_2S emission in WWTPs. Traditionally, odor gas containing H_2S emitted from WWTPs is

first collected by a ventilation system followed by treatments of a wide variety of physical, chemical and biological approaches [4]. In this case, the construction of additional H_2S collection facility and treatment facilities resulted in greater land use and higher investments. In addition, particular chemical agents are also proposed to be added directly into wastewater to control H_2S emission in WWTPs. Sodium hydroxide, magnesium hydroxide [5], and strong oxidants [6] have been applied for the treatment and prevention of H_2S emissions. Furthermore, metal ions can also be used to precipitate sulfides [7,8]. However, these additional agents also incur high costs and are potentially toxic. Recently, Activated sludge recycling (ASR) technology has been presented as a cost-effective approach for H_2S control. ASR technology is the method of recycling aerobic activated sludge from aerated biological reactors or secondary clarifiers to mix with the influents of the WWTP [9]. ASR technology has been successfully applied and proven high removal efficiency for H_2S in recent studies [10].

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Unfortunately, the removal mechanism of H_2S by activated sludge has not been investigated systematically until now. Generally, the removal of sulfides by activated sludge was considered to be a result of biodegradation of sulfides in water [11,12]. However, dissolved oxygen (DO) in the influents of wastewater treatment plants has been reported to be in the range of 0.1 to 0.35 mg/L [13]. It is well known that low DO could greatly hinder the performance of aerobic activated sludge. Therefore, sulfides are hardly to be oxidized thoroughly and rapidly by aerobic activated sludge in wastewater influents. In this study, it was assumed that the adsorption behavior of sulfides onto activated sludge is a very important biological process in ASR technology.

Lately, activated sludge has also been found as an effective adsorbent for dyes [14], heavy metals [15,16], antibiotics [17], and even nanoparticles [18]. Importantly, phosphate adsorption by activated sludge has been reported and researched systematically [19]. Therefore, it is reasonable to consider that other anions in aqueous solution may also be adsorbed onto activated sludge. Microorganisms are abundant in activated sludge and their cell walls consist of various organic compounds, including chitin, lipids, amino acids and other cellular components [20]. These organic compounds on the surface are responsible for the adsorption of particular pollutants by activated sludge.

The aim of this study is to further investigate the removal mechanism of H_2S by activated sludge and evaluate the removal behavior. Firstly, sulfides release experiment was carried on to prove the positive adsorption behavior of sulfides onto activated sludge. Moreover, scanning Electron Microscope (SEM) and X-ray photoelectron spectroscopy (XPS) were conducted to study the effects of mixing sulfides process on the relative content and oxidation state of sulfur element. Furthermore, the thermodynamics and kinetics of sulfides adsorption are studied and given. Finally, we test the effect of temperature, pH, sulfides concentration and agitation speed.

2. Materials and methods

2.1. Preparation of activated sludge and sulfides solution

2.1.1. Collection and pretreatment of activated sludge

Sludge samples were collected from the clarifier of a municipal wastewater treatment plant in Beijing, which used the A^2/O biological treatment process, and were subsequently sealed and stored at 4°C . The activated sludge was mixed with deaerated ultrapure water in 45 mL serum bottles before the experiment. The mixture was set aside for 90 min to remove the residual DO by complete respiration. Then, characteristics of the sludge samples were measured. The moisture content was determined to be 99.72% and the volatile suspended solid (VSS) concentration was set to 3.7 g/L. The concentration of sulfides in the sludge was measured as 0.13–0.51 mg/L, which is taken into consideration and will be subtracted from the concentration of sulfides adsorbed by activated sludge.

2.1.2. Preparation of sulfides solution

$\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ is difficult to weigh because of deliquescence. An aliquot of $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ was dissolved in deaerated ultrapure water and the pH of the Na_2S solution was adjusted to 7.2–7.3 for 1 M HCl. Finally, the Na_2S solution was diluted to the suitable concentration of sulfides and then used as sulfides solution.

2.2. Investigation on mechanism of sulfides removal by activated sludge

2.2.1. Sulfides release experiment

The aim of this particular test is to determine the proportion of adsorption and biodegradation of sulfides. Sulfides solution with an initial concentration of 10 mg/L was prepared for removal and release experiments. Respectively, 30 mL sulfides solution was added into four groups of 44 mL serum bottles. In advance, 14 mL ultrapure water was added to the control bottle, while 4 mL activated sludge and 10 mL ultrapure water were added to the bottles of the experimental groups. Varying de-aeration times of ultrapure water to prepare sulfides solutions, different concentrations of DO were chosen as 0.49, 2.19 and 6.95 mg/L respectively, in experimental groups, while the concentration of DO in the control group was 6.95 mg/L. All of the sulfides solutions in the four groups were mixed with activated sludge for 40 min at certain conditions (initial sulfides concentration 10 mg/L; temperature 25°C ; agitation speed 400 r/min; pH 7.2). Release experiments were performed immediately after the mixture of sulfides and activated sludge. The pH of the solution in all serum bottles was adjusted to 1.0 for 0.1 M HCl. Bottles were set aside for 5 min to ensure complete release of sulfides. Finally, the suspension was extracted with a syringe and filtered through 0.22 μm membranes to analyze the concentration of sulfides in the mixing and release experiments.

2.2.2. Scanning electron microscope/Energy dispersive spectrometer (SEM/EDS) test

In order to observe the interaction of activated sludge and sulfides, the mixing of sulfides and activated sludge was repeated, procedure were identical to the sulfides release experiment, after 10 min of de-aeration, in 0.47 mg/L of DO concentration, 30 mL sulfides solution were mixed with 4 mL activated sludge and 10 mL ultrapure water for 40 min at fixed conditions (initial sulfides concentration 10 mg/L; temperature 25°C ; agitation speed 400 r/min; pH 7.2), then the activated sludge sample before and after the mixing with sulfides solution were further treated and examined by SEM/EDS. Each sample was centrifuged to dispose all solution then dewatered for 48–72 h in freeze dryer (FDU-1100). After the treatment, activated sludge samples were applied evenly to the conductive tape which is attached to the copper plate, then were evenly pressed, followed by sputter-coating of gold, finally observed with SEM (JSM-6460LV) at the magnification of $500\times$ and scanned in EDS (Oxford Instrument). Data are analyzed in INCA-analyzer software.

2.2.3. X-ray photoelectron spectroscopy (XPS) test

The oxidation state of sulfur element in dewatered activated sludge before and after the mixing of sulfides and activated sludge were analyzed by XPS (ESCALab220i-XL). Monochromatic $\text{AlK}\alpha$ (300 W, $h\nu = 1486.8\text{ eV}$) X-rays were used to excite the samples pretreated by the method mentioned in 2.2.2, and the diameter of analysis area was 500 mm. S 2p spectrum was obtained at the pass energy of 100 eV in the fixed analyzer. Finally, the binding energy was calibrated based on the C1s feature at 284.6 eV.

2.2.4. Fourier transform infrared spectroscopy (FTIR) test

1 mg of activated sludge samples before and after mixing with sulfides solution respectively, were freeze dried then mixed with 400 mg of potassium bromide (spectrographic pure), followed by pressing in the pressure of 10 t/cm² to obtain a transparent pellet. Finally, the FTIR spectra were recorded on a FTIR spectrometer (NICOLET6700, ThermoFisher).

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