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Experimental Study of Al-Modified Zeolite with Oxygen Nanobubbles in Repairing Black Odorous Sediments in River Channels

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Abstract: As an extreme phenomenon of water pollution, black odorous water not only causes ecological damage, but also severely restricts urban development. Presently, the in situ remediation technology for sediment from river channels is still undeveloped, and there are many bottlenecks in the key technologies for sediment pollution control and ecological restoration. In this study, three experimental tanks were used to explore the restoration effect of Al-modified zeolite with oxygen nanobubbles on black odorous sediment from the Shichuan River. One of the tanks housed *Typha orientalis* and *Canna indica* L. (TC), another tank housed the same plants and had Al-modified zeolite with oxygen nanobubbles (TC+AMZON), and the last tank was used as a comparison test (CS). The results show that the nitrogen (N) and phosphorus (P) in the sediment are violently released into the surrounding water. However, TC+AMZON could effectively inhibit the release of P. The released amount of soluble reactive phosphorus (SRP) from the pore water in the sediment reached its maximum at 40 d, and the amounts were 122.97% and 74.32% greater in TC and CS, respectively, than in TC+AMZON. However, the released amount of total phosphorus (TP) reached its maximum at 70 d, and the amounts were 260.14% and 218.23% greater in TC and CS, respectively, than in TC+AMZON. TC+AMZON significantly increased the dissolved oxygen (DO) and the oxidation-reduction potential (ORP) of pore water in the sediment in the early stages of the test. At 0 d, the DO content in TC+AMZON reached 10.6 mg/L, which is 112.0% and 178.95% greater than in TC and CS, respectively. The change law of ORP in the sediment is consistent with the DO. TC+AMZON significantly improved the transparency and reduced the content of chlorophyll$_a$ in the upper water and could slightly reduce the N and P content in overlying water. The transparency of TC+AMZON increased by 130.76% and 58.73%, and chlorophyll$_a$ decreased by 55.6% and 50.0% when compared to TC and CS, respectively.

Keywords: Al-modified zeolite; oxygen nanoparticles; black odorous sediments; repairing

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Citation: Guo, C.; Wang, H.; Wei, Y.; Li, J.; Peng, B.; Shu, X. Experimental Study of Al-Modified Zeolite with Oxygen Nanobubbles in Repairing Black Odorous Sediments in River Channels. *Water* **2022**, *14*, 2194. https://doi.org/10.3390/w14142194

Academic Editor: Danny D. Reible

Received: 18 May 2022
Accepted: 6 July 2022
Published: 11 July 2022

1. Introduction

With the development of industrial and agricultural production and the acceleration of urbanization, more and more rivers have been polluted [1]. Rivers become "black and stinky", and they have a serious impact on the lives of residents and the surrounding environment. As an extreme phenomenon of water pollution, black odorous water not only causes ecological damage, but also severely restricts urban development [2,3]. The remediation of black odorous water has become one of the most difficult environmental problems in water protection [4].

Many black odorous water remediation projects experience a strange phenomenon of “remediation every year, black odorous every year”. This is mainly due to the fact that people usually focus on the remediation process and the short-term effects but ignore the good water quality maintenance and the long-term effects [5,6]. How to choose the appropriate treatment technology is a key issue that should be solved urgently [7]. In addition, preventing water repeated water deterioration and achieving the long-term maintenance of good water quality are the keys to completely eliminating urban black odorous water after the remediation [8,9]. At this stage, the main technical bottlenecks are the control of the sediment pollution in the river channel and the reconstruction of healthy river ecosystems.

The primary task is to recognize the causes and to take effective control measures. The main causes of sediment pollution in urban rivers are the input of exogenous pollutants, such as industrial wastewater, domestic sewage, garbage, and non-point source pollution, on both sides of the river. However, the pollutants released from river sediments accelerate the deterioration of water quality [10]. The environmental factors mainly include organic pollutants, nitrogen, phosphorus, iron, manganese, sulfide, and other pollutants [11]. The lack of oxygen and poor fluidity in the water can further accelerate the black odorous sediments [12]. Many effective works about the control of exogenous pollutants have been carried out, and control can be achieved by improving the sewage pipeline network, increasing the efficiency of rainwater and sewage diversion, and centralizing sewage treatment and discharging [13]. However, there are still many bottlenecks in the technology for the treatment of endogenous sediment pollution [14,15]. Therefore, chemical inactivation using phosphorus (P) and nitrogen (N) binders has been increasingly used for the management of internal nutrients in the sediment of degraded bodies of water [16].

Zeolite is an aluminosilicate mineral that widely exists in nature. The main components are SiO_2, Al_2O_3, and iron oxides. Natural zeolite is one of the most abundant reserves and is characterized by its unique pore structure, large specific surface area, strong surface adsorption, and ion exchange. Therefore, it is widely used in the adsorption of ammonia nitrogen and heavy metal ions [17]. However, the removal effect of natural zeolite on anions such as phosphate is poor, and it is affected by many factors such as temperature, particle size, etc. [17,18]. The modification of natural zeolite through different methods can effectively increase the removal efficiency of anions such as phosphate. Lin, J. et al. [19] showed that natural zeolite/hydrochloric acid modified by zeolite and calcite composites can effectively reduce the release of N and P in sediments. The phosphorus passivation effect is increased by the dosage and the small particles, and the effect of adding zeolite is better than that of the calcite [20,21]. Gibbs, M. et al. [22,23] compared the effect of four passivators: modified zeolite Z2G1, phoslock, alum, and allophane. The results show that Z2G1 can effectively inhibit the release of P in sediments and that heavy metal ions do not release into the surrounding water from sediments. In addition, most of the oxygen in river sediment is consumed by reducing substances, and the amount of oxygen that reaches the sediment–water interface is very small. Shi, M. et al. [24] showed that algae-induced anoxia/hypoxia can be reduced or reversed after oxygen nanobubbles (diameter less than 1 μm) are loaded into the zeolite micropores and delivered to the anoxic sediment. The manipulation of microbial processes using the surface oxygen nanobubbles potentially allowed them to serve as oxygen suppliers. Therefore, oxygen nanobubbles have the advantages of good stability and a high oxygen mass transfer rate, which provides great potential for the research and development of precise oxygenation technology at the sediment–water interface of river sediments.

In this study, Al-modified zeolite with oxygen nanobubbles is used to control black odorous sediment pollutants in the river channel through three pilot tests, and aquatic plants are used for restoration. This will provide a new technology for the remediation of black odorous water in urban river channels.

2. Materials and Methods

2.1. Materials

1. Al-modified zeolite with oxygen nanobubbles

Natural zeolite is modified using aluminum salt, and an efficient, low-risk, low-cost, and in situ passivation material for sediment pollution is developed, called Al-modified zeolite (natural zeolite 30% + aluminum salt 15% + Stone powder 55%). The main component of stone powder is $CaCO_3$, and it has a large specific surface area. After the zeolite is modified by aluminum salt, the Al^{3+} is hydrolyzed to form a positively charged $Al(OH)_3$ colloid. Oxygen nanobubbles are loaded into the zeolite micropores that can effectively increase the DO concentration at the sediment–water interface. Thus, it can significantly inhibit the release of phosphorus from the sediment. Al-modified zeolite with oxygen nanobubbles oxidizes the active Fe (Fe^{2+}) in the sediment into iron oxide (Fe^{3+}), which then forms an iron oxide layer on the surface (1 $\pm$ cm) of the sediment, which can inhibit the release of Fe-P and enhance the sediment's P-fixed capacity.

2. Aquatic plants

The aquatic plants *Typha orientalis* and *Canna indica* L. are used, as they have excellent pollutant adsorption ability. Physical–biological–microbial methods are used to optimize the structure of the local water ecosystem and to improve the primary productivity of water and to convert nutrients in water into plant tissues.

2.2. Test Device

In March 2021, three test tanks with the dimensions length $\times$ width $\times$ height = 2 m $\times$ 1 m $\times$ 1 m were constructed in the Fuping pilot test base of Shaanxi Provincial Land Engineering Construction Group Co., Ltd., Weinan city, China. Black and odorous mud was laid in the bottom in a 10 cm thick layer. the mud was taken from the Shichuan River, Fuping County, Shaanxi Province China. Then, about 20 cm of water was injected and was left for about 10 days to discharge chlorine gas. A week later, the aquatic plants *Typha orientalis* and *Canna indica* L. were planted in one of the tanks, then Al-modified zeolite with oxygen nanobubbles was added in a 2 cm thick layer, and the tank was marked as TC+AMZON. The particle size of the zeolite was about 2~3 mm. In the other tank, only *Typha orientalis* and *Canna indica* L. were planted, and this tank was marked as TC. The last tank was used as a comparison test, and it was marked as comparison test (CS). The test process is shown in Figure 1. The test device was completed in May 2021.

(**a**)

(**b**)

Figure 1. *Cont.*

(c)

Figure 1. Test process figures: (**a**) sediment drying; (**b**) sediment filling; (**c**) scene drawing.

2.3. Sample Collection

On 16 May 2021, Al-modified zeolite with oxygen nanobubbles was added into the device. On 26 May, the overlying water and sediment samples were collected. The water samples were taken from 5 cm below the water surface, and the sediment samples were taken from depths within 2 cm below the sediment surface. After the samples were collected, the Al-modified zeolite was supplemented at the sampling point. Water and sediment samples were collected from the three tanks every 10 days in the early stage, and they were collected every 15 or 20 days in the later stages. The collected water and sediment samples were stored at −4 °C, and the analysis was completed within one week. The test indicators and methods are shown in Table 1.

Table 1. Test indicators and methods.

Indicators	Test Methods
Dissolved oxygen (DO)	In situ test
Chemical oxygen demand (COD)	Determination of the chemical oxygen demand—Dichrom method
Transparency	Methods of monitoring and analyzing water and wastewater [20]
NH_4^+-N	Air and exhaust gas—determination of ammonia using Nessler's reagent spectrophotometry
Total nitrogen (TN)	Alkaline potassium persulfate digestion using UV spectrophotometric method
Total phosphorus (TP)	Ammonium molybdate spectrophotometric method
Soluble active phosphorus (SRP)	Analysis of water used in boiler and cooling system—determination of phosphate
Chlorophyll$_a$	Determination of chlorophyll$_a$—spectrophotometric method
Oxidation-reduction potential (ORP)	In situ test

3. Results and Discussion

3.1. N and P of Pore Water in Sediment

The TP and SRP contents of the pore water in the sediment in CS and TC show an increasing trend before 130 d, and they all maintain a high level of P content (Figure 2). This indicates that the P of the sediment is released into the surrounding water as the temperature increases. The released amount of SRP reaches its maximum at 40 d, and it is 122.97% and 74.32% greater in CS and TC than in TC+AMZON at this time, respectively. The released amount of TP reaches its maximum at 70 d, and it is 260.14% and 218.23% greater in CS and TC than in TC+AMZON, respectively. The TP and SRP contents in TC+AMZON increased slightly before 70 days, but both are less than in CS and TC. The main reason for this is that during the anaerobic period in summer, Fe-P from the sediment is reduced to form a large amount of SRP, resulting in the strong release of P into the sediment [25]. However, the addition of the Al-modified zeolite with oxygen nanobubbles makes the sediment locally aerobic and oxidizes Fe (Fe^{2+}) to iron oxide (Fe^{3+}). Then, it forms an iron oxide passivation layer, and Fe^{2+} is converted into Fe^{3+}. SRP/Fe-P is strictly fixed among the sediment and effectively reduces the release of P. However, CS and TC are in an anaerobic environment without the addition of Al-modified zeolite with oxygen nanobubbles, and they have a very high concentration gradient of Fe-P on the sediment surface, so this leads to a strong P release from the sediment [26,27]. When the Al-modified zeolite with oxygen nanobubbles is added to the surface of the sediment in TC+AMZON, the nanobubbles continuously release oxygen and form an oxide layer on the surface of the sediment, which not only reduces the concentrations of P and Fe but also greatly reduces the concentration gradient of P. Thereby, the release of P from the sediment is inhibited. Fortunately, the oxygen nanobubbles in the Al-modified zeolite play an important role. In addition, zeolite has a large specific surface area and strong adsorption [28,29]. The natural zeolite was rich in Al^{3+} after modification with aluminum salt, and the Al^{3+} was hydrolyzed to form an $Al(OH)_3$ colloid, which plays an important role in the adsorption of $PO_4{}^{3+}$ in a water body [30,31]. The Al-modified zeolite could reduce the release of P from the sediment and improve the overlying water quality. Therefore, it achieved the dual purpose of controlling P pollution and preventing eutrophication in overlying water. At the same time, the available P in the pore water was absorbed by the aquatic plants, so the P from the sediment was effectively removed. The in situ passivation of river sediment with phytoremediation is a P pollutant treatment technology with high efficiency, low cost, environmental protection, and aesthetics [32].

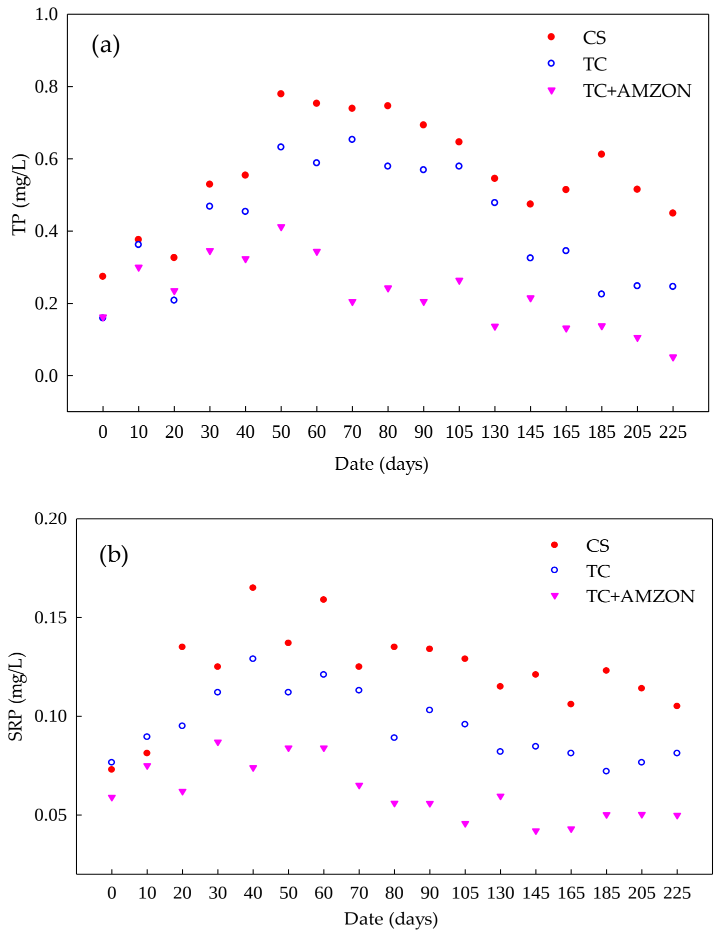

Figure 2. *Cont.*

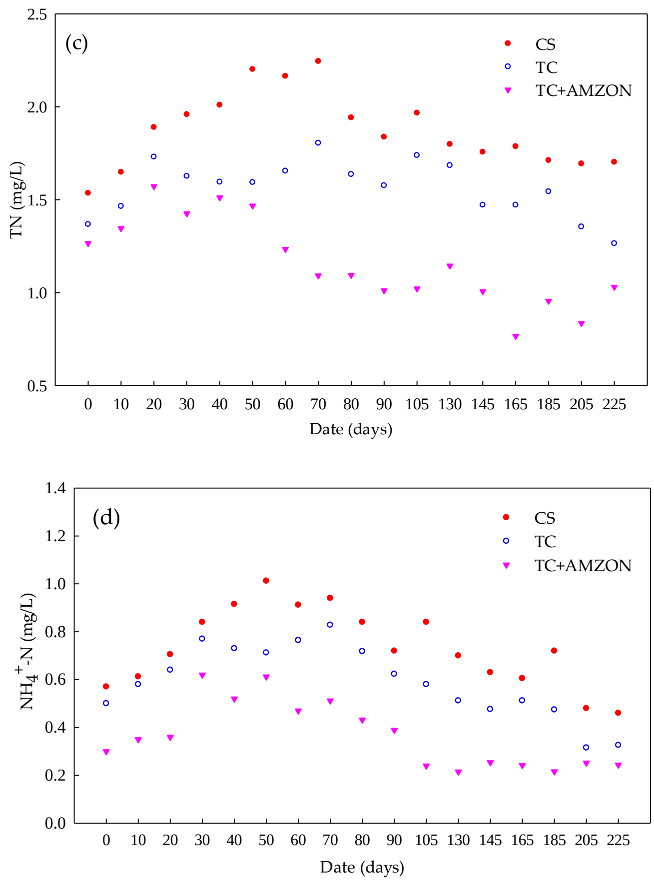

Figure 2. N and P of pore water in sediment: (**a**) TP of pore water in sediment; (**b**) SRP of pore water in sediment; (**c**) TN of pore water in sediment; (**d**) NH_4^+-N of pore water in sediment.

The TN and NH_4^+-N contents of the pore water from the CS and TC sediments also first show an increasing trend and then a decreasing trend. The influence of TC+AMZON on the TN and NH_4^+-N contents is great, and they are significantly lower than those in CS and TC. This indicates that the sediment also releases N into the surrounding water and that the Al-modified zeolite with oxygen nanobubbles inhibits N release. Studies have shown that nanobubbles have negative charges at the gas–liquid interface that can interact with positively charged pollutants in the water, and the free radicals and vibration waves that are generated can promote the removal of pollutants [33]. Therefore, the pollutants discharged from urban non-point source pollution to rivers and lakes can be repaired by in situ Al-modified zeolite + phytoremediation technology, which can achieve the effect of pollutant control and can beautify the city. It is the most effective technical means at present.

3.2. DO and ORP of Pore Water in Sediment

The addition of Al-modified zeolite with oxygen nanobubbles can significantly increase the dissolved oxygen (DO) content in the pore water of sediment (Figure 3). During the experimental period, the DO in CS was kept between 2.9 and 4.3 mg/L, and it varied from 5.3 to 7.5 mg/L in TC. However, the DO in TC+AMZON was kept at 6.2–10.6 mg/L. Before 60 days, the DO content in TC+AMZON was significantly greater than that in CS and TC. On day zero, the DO content in TC+AMZON reached 10.6 mg/L, which is 178.95% and 112.0% greater than that in CS and TC, respectively. With the continuation of the experiment, the DO content of the pore water in TC+AMZON gradually decreased, which was mainly caused by the continuous oxygen consumption of the reducing substances and the microbial activities in the sediment. Sixty days later, the DO concentration in TC+AMZON decreased below 5.5 mg/L, which is consistent with TC, and gradually became stable. The results show that the Al-modified zeolite with oxygen nanobubble has a good ability to increase the oxygen at the sediment–water interface. Therefore, the addition of Al-modified zeolite with oxygen nanobubbles is beneficial to increase the DO content in the pore water in sediment.

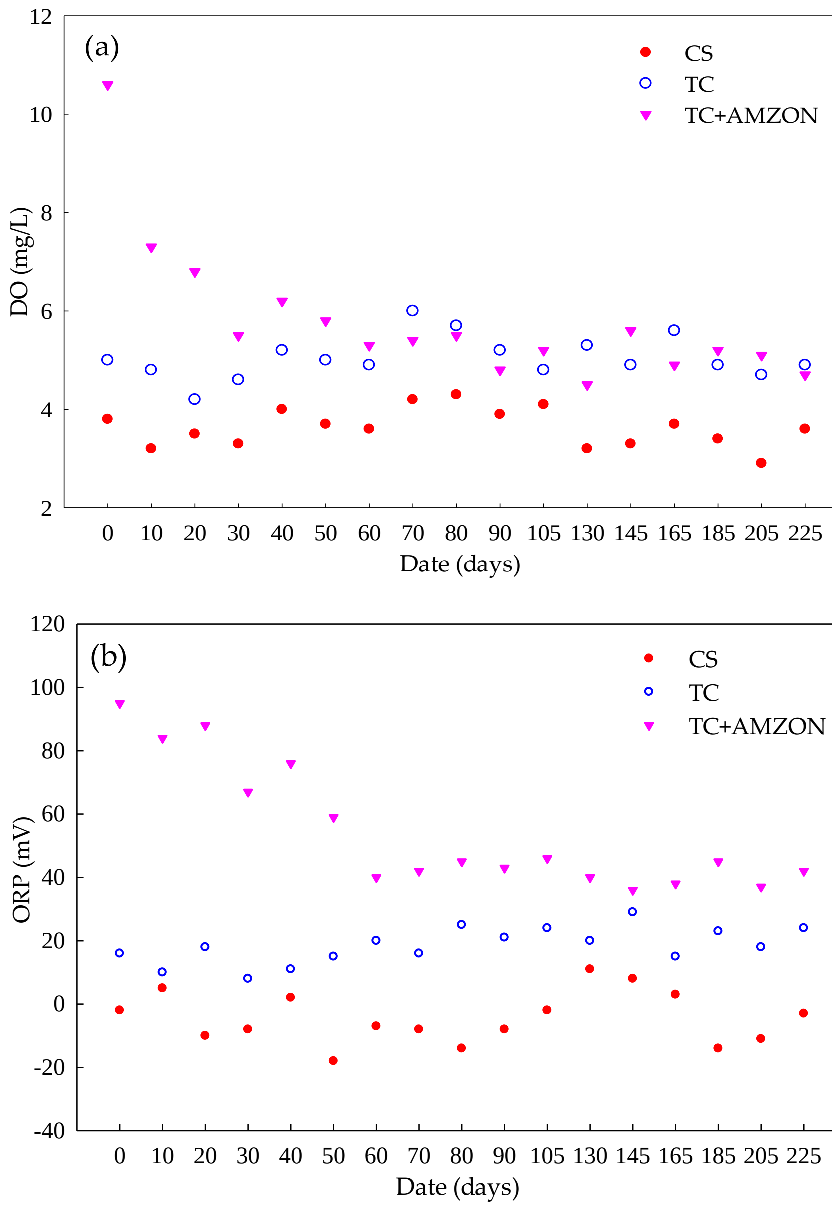

Figure 3. DO and ORP of pore water in sediment: (**a**) DO of pore water in sediment; (**b**) ORP of pore water in sediment.

The oxidation-reduction potential (ORP) on the sediment surface of CS varied from −35 to 20 mV and was kept between 5 and 35 mV in TC; however, the ORP in TC+AMZON varied from 12 to 95 mV. Therefore, the ORP of pore water in TC+AMZON significantly increased. Moreover, the change process of the ORP of the sediment was consistent with the DO, and this indicates that the distribution of the DO content of the sediment affects the ORP, which is consistent with the results of Shi et al. [24]. The transformation and diffusion of most of the dissolved substances in sediments are affected by their ORP.

3.3. N, P and COD in Overlying Water

It can be seen in Figure 4 that the TP content in the overlying water decreases with the experimental time. The TP content in the three tanks is in the order of CS > TC > TC+AMZON, and TP contents in TC and TC+AMZON are all smaller than the TP content in CS. This shows that the release of P from the sediment will not increase the P content in the overlying water. The main reason for this is that the Al-zeolite particles rapidly adsorb the dissolved active P from the sediment. Relevant studies have shown that aluminum ions of the modified zeolites are hydrolyzed to form positively charged $Al(OH)_3$ colloids, which can strongly adsorb negatively charged bacteria and ions in water [34,35], such as ${PO_3}^{4-}$. At the same time, $Al(OH)_3$ colloids are a type of light-weight suspended matter and are not easy to precipitate [36,37]. However, the $Al(OH)_3$ can increase its mass after adsorbing ions in water and gradually settles to the bottom of the water, reducing the possibility of resuspension. In addition, the hydrolyzed product $Al(OH)_3$ provides bridging adsorption to adsorb suspended solids in water [38,39]. Al^{3+} is hydrolyzed to form a high molecular polymer with a linear structure. One side of the high molecular polymer can adsorb a particle far away, and the other side extends into the water to absorb another particle. The particle is bridged by polymer adsorption, which let it gradually become bigger and bigger [40]. With the increase in the particles adsorbed by Al^{3+}, it gradually moves towards the bottom of the water, and it absorbs the suspended particles in the water during the sinking process [32]. After the colloid settles to the surface of the sediment, a covering layer is formed upon the surface of the sediment to prevent the pollutants of the sediment from being released to the overlying water.

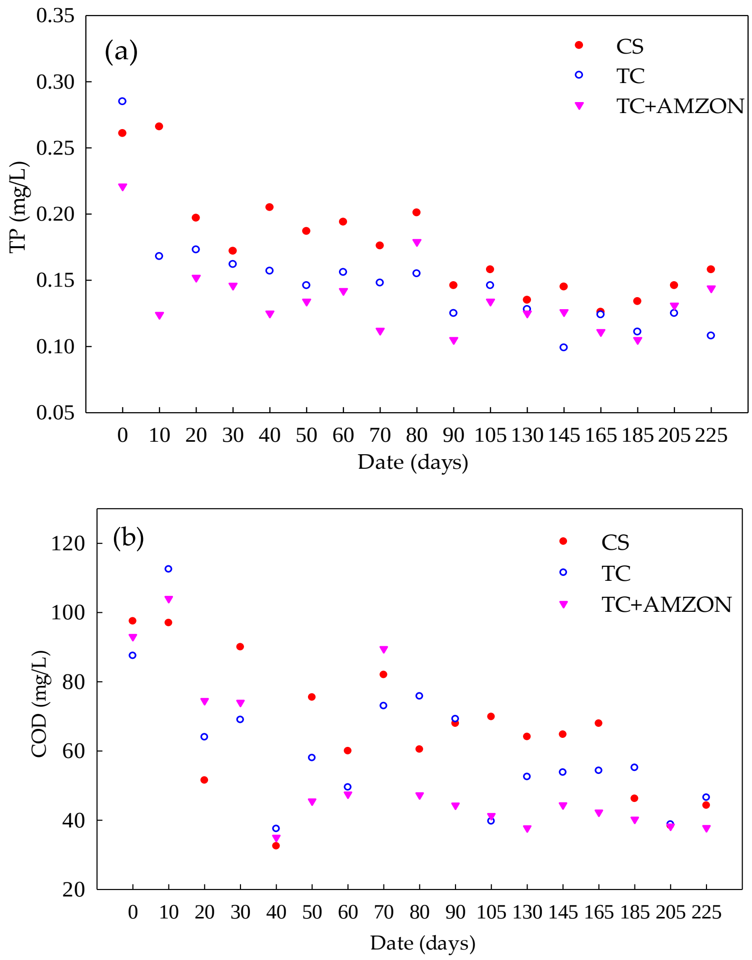

Figure 4. *Cont.*

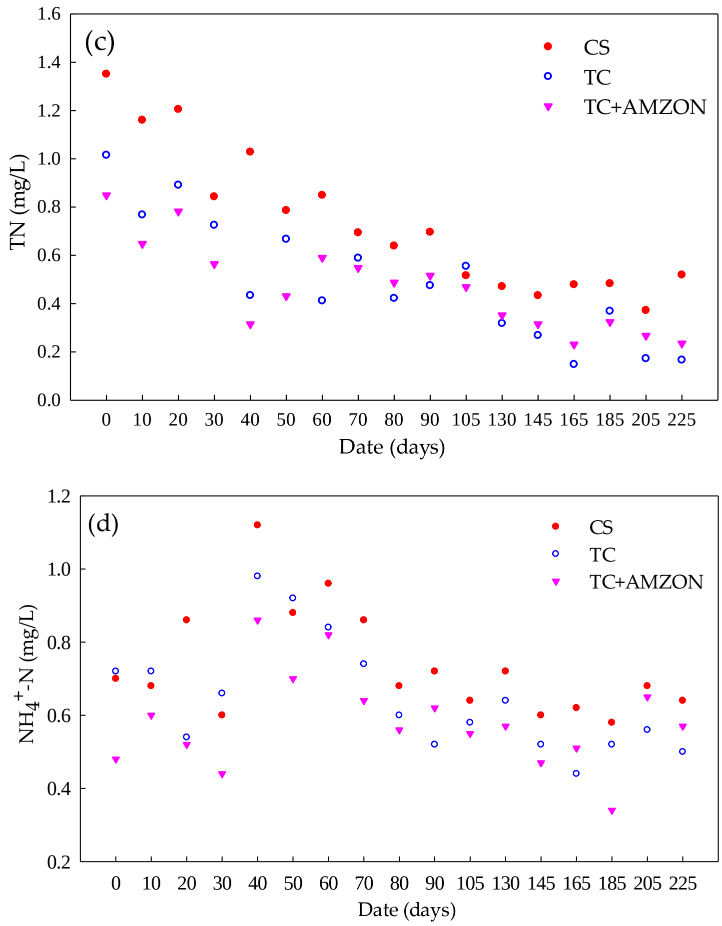

Figure 4. N, P, and COD in overlying water: (**a**) TP in overlying water; (**b**) COD in overlying water; (**c**) TN in overlying water; (**d**) NH_4^+-N in overlying water.

The TN content in the overlying water shows a decreasing trend with the test time, and the NH_4^+-N first shows an increasing trend and then decreases gradually. The TN and NH_4^+-N contents in TC and TC+AMZON are all smaller than in CS. Before 60 days, the contents of TN and NH_4^+-N in TC+AMZON are slightly lower than those in TC, and the differences gradually decreased over the 60 days. The main reason is that the zeolite has an adsorption effect on NH_4^+-N, which reduces the N content in the overlying water. In addition, the absorption of available N by aquatic plants can also reduce the N content in water. Therefore, when using Al-modified zeolite to remediate pollutants from sediment, it is necessary to plant aquatic plants to absorb the pollutants of sediment and water [41].

Al-modified zeolite has little effect on COD in the overlying water. In the early stage of the experiment, there was a minor difference in the COD in the overlying water among the three tanks. However, the COD content of the overlying water in TC+AMZON and TC is slightly lower than that in CS in the later stage. This is mainly because of the adsorption of soluble organic pollutants by the plants that reduce COD content. Therefore, the application of Al-modified zeolite has no obvious improvement effect on the COD in black odorous water.

3.4. Transparency and Chlorophylla in Overlying Water

It can be seen from Figure 5 that the transparency of the overlying water in TC+AMZON and TC gradually increases with the experimental time, while it first shows a decreasing trend and then remains stable in the later period in CS. The difference is even bigger 60 days later (Figure 5). According to statistics, the transparency in TC+AMZON remains at 17.6–30 cm 60 days later and varies from 15.5 to 22.4 cm in TC. However, the transparency in CS remains at 11.6–15.7 cm. Thus, compared to CS and TC, the transparency of the overlying water in TC+AMZON is increased by 130.76% and 58.73%, respectively, at the same sampling time. Therefore, the application of Al-modified zeolite with oxygen nanobubbles has a significant effect on water purification.

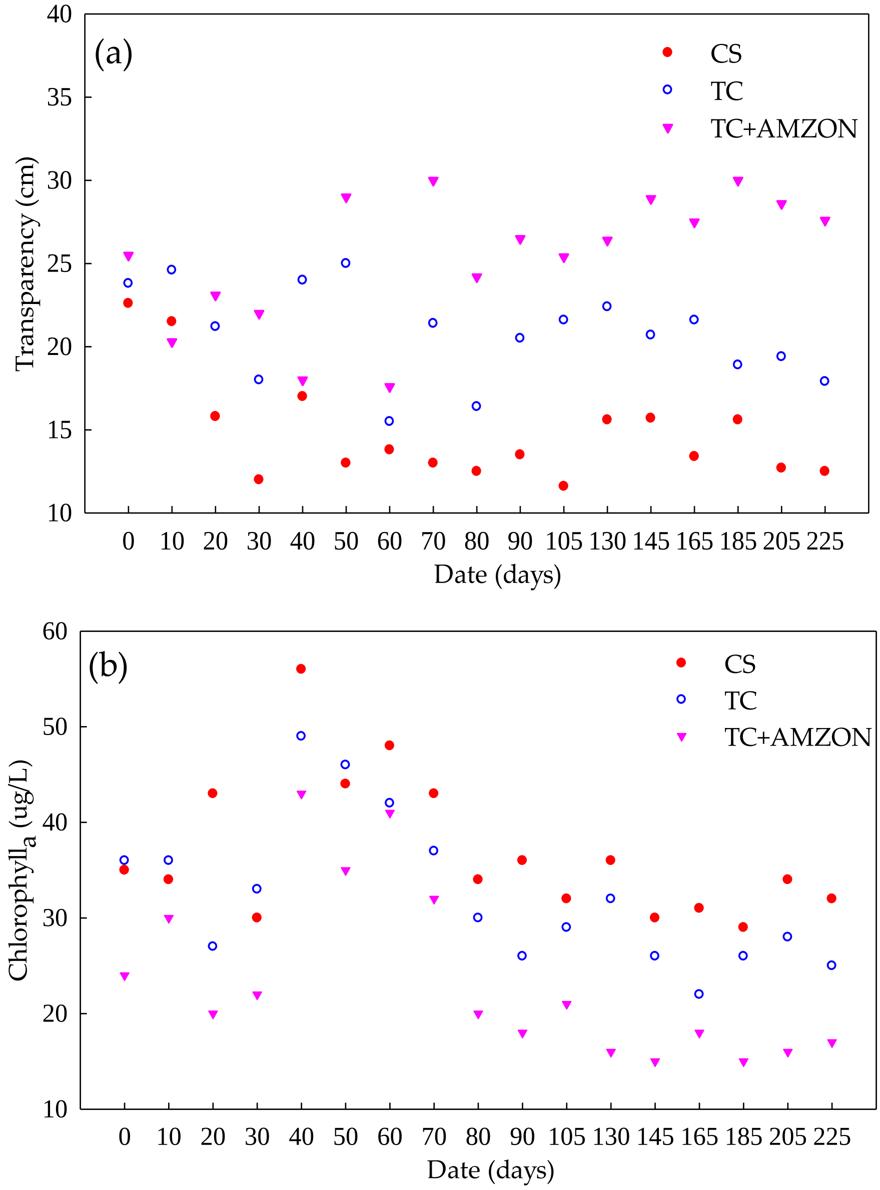

Figure 5. Transparency and chlorophyll$_a$ in overlying water: (**a**) transparency in overlying water; (**b**) chlorophyll$_a$ in overlying water.

The content of chlorophyll$_a$ in the overlying water generally increased first and then decreased before stabilizing. Tt began to increase 30 days later and reached its maximum value 50 days later. The main reason for this is that the experiment started at the end of May. Thirty days later, the temperature was high. There were more prokaryotic blue-green algae (cyanobacteria) and eukaryotic algae in the test tanks, but no green plants were visible. Algae can synthesize organic substances through photosynthesis, which converts light energy into chemical energy, thereby increasing the content of chlorophyll$_a$ in the water [4,42]. Eighty days later, the content of chlorophyll$_a$ in the overlying water in TC+AMZON was significantly less than in TC and CS. The content of chlorophyll$_a$ in TC+AMZON remained between 15 and 21 μg/L but varied from 22 to 32 μg/L and from 29 and 36 μg/L in TC and CS, respectively. The content of chlorophyll$_a$ in TC+AMZON decreased by 50.00% and 55.56% when compared with TC and CS at the same sampling time. This shows that the content of chlorophyll$_a$ in water can be reduced by the addition of Al-modified zeolite with oxygen nanobubbles. The chlorophyll$_a$ content in the three tanks gradually became stable over 105 days.

4. Conclusions

Al-modified zeolite with oxygen nanobubbles was used to remove river sediment in this study, and it demonstrated that:

1. Sediment in the river channel releases P strongly to the surrounding water, but N has a certain release rate. However, the addition of Al-modified zeolite with oxygen nanobubbles can inhibit the release of P and N, and the effect is very obvious.
2. The addition of Al-modified zeolite with oxygen nanobubbles increased the DO and ORP content in the pore water significantly in the early stage of the test. Sixty days later, the DO content in TC+AMZON was reduced to below 5.5 mg/L. The ORP variation in the pore water was consistent with that of the DO.
3. The amounts of P and N released from sediment do not increase in overlying water. There was a minor difference in the TN, TP, and NH_4^+-N contents in the overlying water in TC+AMZON and TC, and their contents are all smaller than those in CS. The addition of Al-modified zeolite with oxygen nanobubbles has a small effect on the COD of the overlying water. In the early stage of the experiment, the difference in the COD content in the three tanks is small; however, it is less in TC+AMZON and TC than in CS in the later stage.
4. The transparency of the overlying water is significantly improved by the addition of Al-modified zeolite with oxygen nanobubbles. Forty days later, the difference in the transparency in TC+AMZON is more and more obvious compared to that of TC and CS. Moreover, the addition of Al-modified zeolite with oxygen nanobubbles can reduce the content of chlorophyll$_a$ in the overlying water. Eighty days later, the chlorophyll$_a$ content in the overlying water in TC+AMZON is significantly less than that in TC and CS.

Author Contributions: C.G., H.W. and J.L. conceived the experiments and analyzed the results; Y.W. and B.P. conducted the experiments; X.S. analyzed the data. All authors have read and agreed to the published version of the manuscript.

Funding: This research is financially supported by the Key Research and Development Program of Shaanxi (2022ZDLSF-06-04 and 2020SF-420), the project of Shaanxi Provence Land Engineering Construction Group (DJNY2022-26), and the National Natural Science Foundation of China (No. 51879215).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: The authors declare that the experiments are not involving human participants (including the use of tissue samples) in this paper.

Data Availability Statement: The authors declare that the datasets generated during and analyzed during the current study are not publicly available due to the data being confidential and the basis for further research but are available from the corresponding author on reasonable request.

Conflicts of Interest: The authors declare no conflict of interest.

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