

# Investigate the Synergistic Mechanism of Nitrogen/sulfur co-doped Carbon and Its Application in the Remediation of Heavy Metals

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## Abstract:

Heavy metal pollution has become a serious global threat, causing great damage to the environment and human health. Nitrogen/sulfur co-doped biochar(NBCs) is of significant importance for the remediation of heavy metal pollution. This paper aims to deeply explore the synergistic mechanism of NBCs in heavy metal remediation. It has been found that NBCs can optimize the pore structure and surface chemical properties of biochar, and significantly improve the adsorption efficiency and stability of heavy metals. This paper also systematically reviews various synthesis processes and modification mechanisms of NBCs, and explores the oxidative precipitation and chemical fixation mechanisms of NBCs in the system of synergistic oxidants. However, existing research still faces challenges such as unclear synergistic remediation mechanisms for multiple metals, insufficient long-term stability of the carbon skeleton, and lack of verification for large-scale applications. In the future, new preparation processes need to be developed to promote the application of NBCs in complex environments, providing theoretical support and practical reference for the development of green and sustainable remediation technologies.

**Keywords:** NBCs, heavy metal remediation, synergistic mechanism, carbon neutrality.

## 1. Introduction

Heavy metal pollution (such as cadmium and lead) has become an urgent threat to the global ecosystem and human health due to its persistent high toxicity, difficulty in degradation, and bioaccumulation. Relevant

studies have pointed out that approximately 14% to 17% of farmland globally is contaminated by toxic metals, severely affecting agricultural production and food security. It is estimated that 900 million to 1.4 billion people live in areas with high pollution risks [1]. Cadmium (Cd) and Lead (Pb) are listed as priori-

ty controlled pollutants due to their strong mobility, which makes them easily transfer from soil to the food chain, and the high difficulty of remediation. However, the remediation of soil contaminated by Cd and Pb is undoubtedly an important and extremely challenging project.

Traditional physicochemical remediation technologies (such as chemical precipitation, ion exchange, and electrochemical treatment) have certain effects, but they have problems such as high costs and secondary pollution [2]. In recent years, biochar has been regarded as an ideal material for heavy metal pollution remediation due to its low cost, unique porous structure, and environmental friendliness. However, the original single biochar material faces problems such as low adsorption capacity for Cd/Pb and insufficient selectivity [3], so it is urgently necessary to improve its performance through functional modification. The nitrogen/sulfur co-doping technology provides a new idea for the application of biochar in heavy metal pollution remediation, and significant progress has been made in the synthesis process and mechanism analysis in recent years. For example, Liu et al. [4] pointed out that NBCs not only enhances the adsorption efficiency of biochar for heavy metals (Pb, Cd), but also improves its stability and reusability, showing good application prospects; Qi et al. [5] prepared porous biochar through N, S co-doping for the removal of nickel ions by capacitive deionization, exhibiting excellent adsorption performance and application potential; in addition, Lai et al. [6] modified biochar through N, S co-doping, significantly enhancing its degradation performance of organic pollutants when co-activating  $H_2O_2$  with trace amounts of  $Fe^{3+}$ , showing a higher reaction rate and better environmental adaptability. Although existing studies have reported the optimization effect of nitrogen/sulfur co-doping on the performance of biochar, its synergistic mechanism in heavy metal remediation still needs in-depth exploration. At the same time, most existing studies focus on single heteroatom modification or the remediation of a single heavy metal, and there is a lack of systematic research on the synergistic mechanism of nitrogen/sulfur co-doping in complex environments polluted by multiple heavy metals.

This paper aims to systematically summarize and analyze the application of NSBCs in heavy metal pollution remediation and its synergistic mechanism. Based on existing experimental data and theoretical calculations by predecessors, this paper compares and analyzes the remediation effect of NBCs in actual polluted environments. Through the above research methods, this paper hopes to provide theoretical support for the research and development of efficient and low-cost heavy metal pollution control technologies and promote the high-value utilization of biomass resources under the global strategy of “carbon neutrality”.

## 2. Synthetic Research on the Synergistic Mechanism of Nitrogen/Sulfur Co-doping in Biochar

### 2.1 Doping process

The heteroatom doping process is the core technology for regulating the physical and chemical properties of biochar, which is mainly divided into two methods. Firstly, the in-situ doping process (utilizing the inherent elements of biochar) means that by optimizing the pyrolysis conditions, heteroatoms such as N and S contained in the biomass itself are retained and directly embedded into the carbon skeleton. For example, Li et al. [7] prepared nitrogen-doped porous coral-like biochar by the in-situ pyrolysis method, significantly increasing the specific surface area (up to  $1693\text{ m}^2/\text{g}$ ) and adsorption capacity of the biochar, and a low nitrogen source ratio is conducive to the development of the pore structure. Secondly, the exogenous doping process (introducing external heteroatoms) means that external heteroatoms are introduced into the biochar skeleton through methods such as chemical impregnation and vapor deposition. For example, Yu et al. [8] prepared shrimp shell biochar by exogenous P and S doping and found that P doping enhanced the electron conduction ability and specific surface area of the biochar, and realized the selective degradation of organic pollutants through a non-radical electron transfer pathway. In addition, several emerging composite processes of heteroatom doping have emerged in the scientific community in recent years. For example, the microwave pyrolysis and nitrogen-sulfur co-doping strategy proposed by Chen et al. [9] successfully prepared biochar (ASBC-800-0.5) with high microporosity and abundant functional groups. Its  $CO_2$  adsorption performance is significantly better than that of traditional materials, providing new ideas for the development of low-cost carbon capture technologies.

### 2.2 Regulation of physical properties by nitrogen/sulfur doping

#### 2.2.1 Hierarchical Pyrolysis Process for the Construction of Porous Structures

The construction of hierarchical pores is the core mechanism for NBCs to improve their adsorption performance. N/S doping forms a synergistic hierarchical pore structure of micropores, mesopores and macropores by regulating the release of volatile components and the shrinkage of the carbon skeleton during the pyrolysis process. Specifically, the mechanism of nitrogen doping lies in that nitrogen-containing functional groups (such as pyridine

nitrogen) produce gases like  $\text{NH}_3$  and  $\text{HCN}$  during pyrolysis. These two gases react with the carbon skeleton at high temperature through the reaction  $\text{C} + 2\text{NH}_3 \rightarrow \text{CH}_4 + \text{H}_2 + \text{N}_2$ , which helps to selectively etch the  $\text{sp}^3$  hybridized carbon regions and form micropores ( $< 2\text{nm}$ ). The effect of sulfur doping is that sulfides (such as thiophene sulfur) react with  $\text{CO}_2$  at high temperature ( $> 600^\circ\text{C}$ ) through the oxidation reaction  $\text{C-S-C} + 3\text{CO}_2 \rightarrow \text{SO}_2 + 4\text{CO}$ . When the generated  $\text{SO}_2$  gas escapes, it expands the spacing between carbon layers and forms mesopores ( $2 - 50\text{nm}$ ). For example, Chen et al. [9] found that the nitrogen (N) and sulfur (S) co-doped biochar (ASBC) prepared by the microwave pyrolysis process balances the pore structure of the biochar, and enhances the adsorption performance of the biochar through acid-base interaction and electron transfer mechanisms. However, as pointed out by Cao et al. [10], changes in temperature and time during the pyrolysis process will affect the formation and distribution of pores, and under the conditions of high temperature and long-term pyrolysis, the ash in the sludge may soften and block the pores, resulting in a decrease in the size and number of pores. Therefore, multiple methods are needed for optimization in the future. Various methods have a regulatory effect on the pore structure: Zhang et al. [11] found that slow pyrolysis is more conducive to the formation of micropores, and Wu [12] further achieves precise control of the pore size distribution through the template method. In addition, Ruan et al. [13] use carbon nanotube

composites to improve the orderliness of the pore structure and the specific surface area, showing superior environmental remediation potential.

### 2.2.2 Optimization of Nitrogen/Sulfur-Carbon Framework Stability

Although nitrogen- and sulfur-doped carbon materials exhibit broad application prospects due to their excellent properties, in practical applications, the carbon skeletons after nitrogen/sulfur doping still face the problem of insufficient stability. For example, Xu et al. [14] pointed out that for nitrogen/sulfur co-doped magnetic biochar (NSM-BC), under conditions of high-concentration electrolyte or long-term reaction, the structural integrity of the carbon skeleton may be damaged, resulting in a decrease in catalytic performance. Therefore, it is crucial to optimize the stability of the nitrogen/sulfur-carbon skeleton. In recent years, researchers have proposed the following optimization strategies: Zhang et al. [15] significantly improved the stability and catalytic activity of the carbon skeleton through an in-situ nitrogen doping method. As shown in Table 1, this is mainly attributed to the introduction of a higher content of pyrrole nitrogen into the carbon skeleton; Wang et al. [16] prepared two-dimensional graphene-like nitrogen/sulfur co-doped porous carbon nanosheets and found that this hierarchical pore structure can provide good physical support for the composite material and increase the overall stability of the material.

**Table 1. Nitrogen configuration in different catalysts**

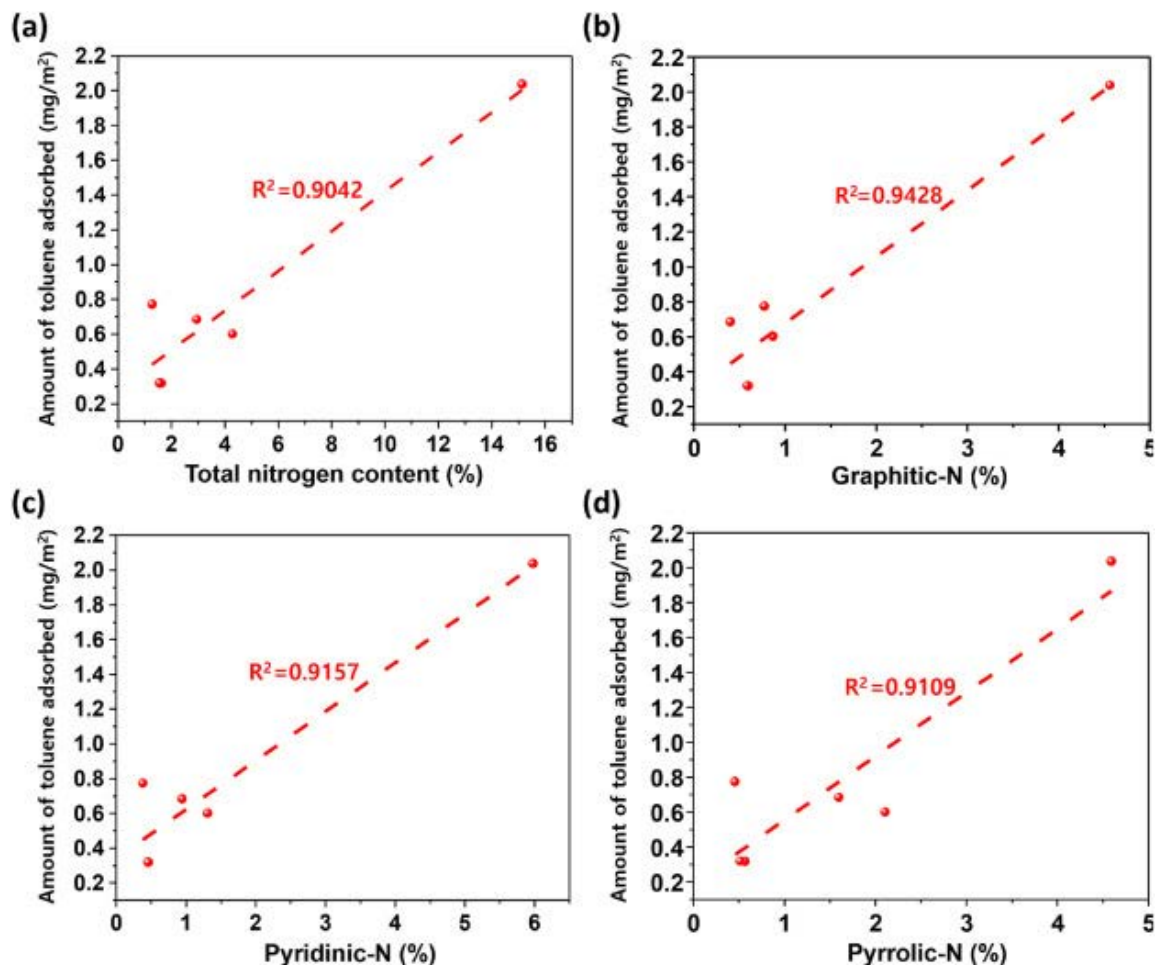
samples	% of total N 1s		
	N-6	N-5	N-Q
Y-NC	26	56	18
Y-NC after	23	31	46
H-NC	50	34	16
H-NC after	25	33	42
AC	29	43	28

## 2.3 Regulation of Chemical Properties by Nitrogen/Sulfur Doping

### 2.3.1 Chemical State Analysis of Pyridinic Nitrogen and Thiophenic Sulfur

Pyridine nitrogen can serve as active sites on the surface of biochar and participate in processes such as adsorption and catalysis. For example, Zaman et al. [17] prepared biochar rich in pyridine nitrogen. Through comparison, it was found that the presence of pyridine nitrogen significantly improved the adsorption capacity of biochar (Fig.

1), and its adsorption mechanism includes electrostatic attraction and coordination. At the same time, thiophene sulfur may also be active on the surface of biochar, especially in redox reactions. For instance, Cui et al. [18] found through research that the structure of thiophene sulfur can promote the activation of peroxymonosulfate (PMS) during the oxidation process, thereby increasing the degradation efficiency of organic pollutants (such as paracetamol).



**Fig. 1. Correlation analysis between the adsorbed amount of toluene per specific surface area and total nitrogen or nitrogen species[17]**

### 2.3.2 The Impact of Nitrogen/Sulfur Doping on Electronic Structure

The electronegativity of nitrogen atoms is different from that of carbon atoms. Its doping will change the electron cloud distribution of biochar, causing the electron density on the surface of biochar to change. This adjustment of the electronic structure can enhance the electrostatic interaction between biochar and heavy metal ions. For example, Kasera et al. [19] found through research that incorporating nitrogen into the biochar matrix will change the overall electronic structure of biochar, which is beneficial to the interaction between nitrogen-doped biochar and heavy metal pollutants. The valence electron structure of sulfur atoms is different from that of carbon. After doping, it will also change the electronic structure of biochar. For example, Seyedi et al. [20] modified biochar with sulfur nanoparticles (SNPs@BC) to provide a low-cost adsorbent for cadmium and lead, which can be used as an efficient and environmentally friendly adsorbent for the removal of heavy metals.

## 3. Study on the Mechanism of Nitrogen/Sulfur Co-doped Biochar in the Remediation of Cd/Pb

### 3.1 The adsorption effect of the co-doped biochar on Cd and Pb

In recent years, researchers have found that co-doped biochar exhibits unique advantages and multiple action mechanisms in the adsorption of Cd and Pb, which are mainly as follows: (1) Improving the adsorption efficiency of biochar. Nitrogen doping can increase the alkaline functional groups on the surface of biochar (such as amino groups, pyridine nitrogen, and pyrrole nitrogen), providing a better chemical environment for the mercapto groups introduced after sulfur doping, enabling it to interact more effectively with  $\text{Cd}^{2+}$  and  $\text{Pb}^{2+}$ , thus enhancing the adsorption effect [21]. (2) Improving the specific surface area and pore structure. Nitrogen/sulfur co-doping can change the thermal stability and surface chemical



properties of biochar, thus forming a more developed pore structure during the preparation process. At the same time, co-doped biochar usually has a higher specific surface area and pore volume, which provides more adsorption sites for  $\text{Cd}^{2+}/\text{Pb}^{2+}$  ions, thereby improving the adsorption efficiency [22]. (3) Improving the adsorption stability. The chemical bonds and complexes formed by nitrogen/sulfur co-doping have higher stability, which can enhance the stability and long-term effectiveness of biochar in adsorbing Cd and Pb under different environmental conditions (such as changes in pH value and redox potential) [23].

### 3.2 Study on the degradation process of the synergistic oxidant

The addition of synergistic oxidants (such as  $\text{H}_2\text{O}_2$  and persulfate) can activate the active sites on the surface of co-doped biochar, generate free radicals such as  $\bullet\text{OH}$  and  $\bullet\text{O}_2^-$ , and promote the oxidative precipitation and chemical fixation of heavy metals. Specifically, in the  $\text{H}_2\text{O}_2$  synergistic system, researchers have found that  $\text{H}_2\text{O}_2$  undergoes heterolytic decomposition at the N/S sites to generate  $\bullet\text{OH}$  and  $\bullet\text{O}_2^-$  free radicals. These free radicals attack heavy metal ions to form hydroxide precipitates (cadmium hydroxide, lead hydroxide). At the same time, the acid-base buffering capacity of the N/S functional groups maintains the pH of the system at 5–7, avoiding the competition between  $\text{H}^+$  and heavy metal ions for adsorption sites under strong acid conditions [24]. In the persulfate synergistic system, the  $\text{S}=\text{O}$  groups generated by sulfur doping act as Lewis acid sites and combine with the peroxy bond ( $-\text{O}-\text{O}-$ ) of PDS, reducing the activation energy. Meanwhile, the lone pair electrons of pyridine nitrogen are transferred to the O atoms of PDS, promoting the cleavage of the peroxy bond and generating highly active sulfate radicals [25].

### 3.3 The process of remediating heavy metal pollution through nitrogen/sulfur complexation

Nitrogen/sulfur co-doping introduces abundant functional groups (such as amino groups, imino groups, sulfhydryl groups, etc.) into biochar. The lone pair electrons in these functional groups can form coordination bonds with the empty orbitals of  $\text{Cd}^{2+}$  and  $\text{Pd}^{2+}$  to generate stable complexes, significantly enhancing the ability to capture heavy metals, etc. [9]. On the one hand, Li et al. [26] found through research that the micro-nano nitrogen-containing straw biochar is rich in nitrogen-containing functional groups, such as amino groups on its surface. These functional groups can complex with  $\text{Pb}^{2+}$  and other ions, thus enhancing the adsorption performance for  $\text{Pb}^{2+}$ . On the other hand, Fan et al. [27] pointed out that after sulf-

hydryl functional groups are successfully introduced on the surface of biochar, the sulfhydryl groups can undergo complexation reactions with heavy metal ions, increasing the adsorption capacity for Pb and Cd.

## 4. Performance Improvement and Case Analysis of Nitrogen/Sulfur Co-doped Biochar

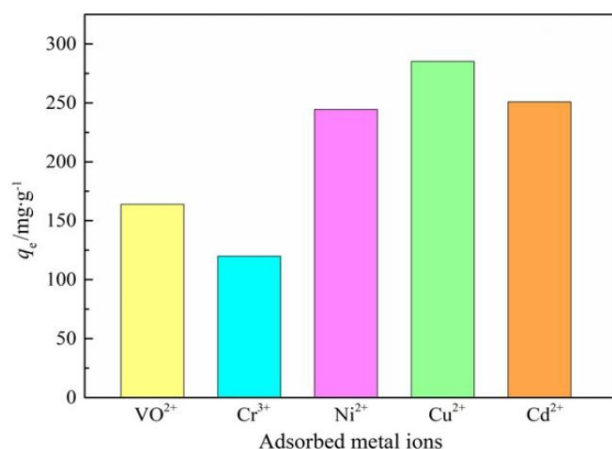
### 4.1 Methods for improving the remediation effect

At present, the remediation effect of NBCs can be improved through the following methods: (1) Optimizing the selection of raw materials: According to the research of Zhao et al. [28], sewage sludge (FO) has the strongest adsorption capacity for heavy metals, followed by agricultural waste (such as rapeseed LO and corn ZO), while wood (poplar CO) has a relatively weak adsorption capacity. (2) Optimizing the preparation conditions: Zhao et al. [28] found that the biochar prepared by slow pyrolysis can change the pore structure, functional groups and mineral content of the raw materials, thus improving the adsorption effect. (3) Utilizing surface characteristic adsorption: Functional groups such as hydroxyl groups ( $-\text{OH}$ ) and carboxyl groups ( $-\text{COOH}$ ) present on the surface of biochar can undergo complexation reactions with heavy metal ions. Biomass and biochar with a higher content of carboxyl groups have a better adsorption effect on heavy metals [28]. (4) Paying attention to ion exchange adsorption: The research of Zhao et al. [28] found that heavy metal ions can be removed through ion exchange with mineral particles (such as Ca, K, Mg, etc.) in the biochar. Especially in a multi-metal system, cation exchange is the main adsorption mechanism, and its influence is greater than that of complexation.

### 4.2 Case Analysis of the Remediation Effect

Although there are few application cases of NBCs in the remediation of heavy metal-contaminated soil at present, the results obtained from relevant studies have shown great potential. For example, through adsorption experiments, Shi et al. [29] found that the adsorption effect of NBCs on various heavy metal ions is significantly better than that of commercial activated carbon. The adsorption capacity for different heavy metals is shown in Fig. 2, indicating great potential in the treatment of heavy metal pollution. The research by Polyakov et al. [30] has shown that nitrogen/sulfur doping not only increases the specific surface area and pore structure of biochar, but also improves the activity of surface functional groups, thus

significantly enhancing its adsorption capacity for heavy metals in the soil. Ghassemi-Golezani et al. [31] have found through research that NBCs significantly reduces the mobility and bioavailability of heavy metals in the soil, and at the same time promotes plant growth and reduces the toxic effects of heavy metals on plants.



**Fig. 2. Adsorption of a series of metal ions by EWC ( $C_0 = \sim 100 \text{ mg} \cdot \text{L}^{-1}$ , dosage =  $0.2 \text{ g} \cdot \text{L}^{-1}$ ) [30]**

## 5. Challenges and Prospects

At present, the treatment of heavy metal pollution through co-doped biochar is a very promising trend. However, the following challenges still remain: Firstly, in the actual polluted environment where multiple heavy metals coexist, the mechanism of competitive adsorption is unclear, and the mechanism of synergistic remediation of multiple metals is not well-defined. Secondly, the long-term stability of the carbon skeleton is insufficient, and its tolerance to extreme environments is poor, resulting in the loss of heteroatoms. Finally, there is a lack of verification of large-scale applications, there are gaps in engineering technologies, and it is difficult to ensure the actual remediation effect.

In order to better address the above challenges, in-depth research can be carried out in the future as follows. On the one hand, by combining in-situ characterization with multi-technology integration, the mechanism analysis can be deepened to better meet the needs in different remediation scenarios. On the other hand, through composite reinforcement or modification of heteroatom immobilization, the mechanical strength of the carbon skeleton can be enhanced. At the same time, it is necessary to explore new doping processes to reduce costs and increase efficiency, and to promote the large-scale application of co-doped biochar in practice. Finally, a long-term assessment of the environmental effects of co-doped biochar remediation can be established to better understand and optimize its

remediation effect.

Through the above research, NBCs is expected to further develop and be applied in the field of heavy metal pollution remediation, providing a strong guarantee for the realization of sustainable development.

## 6. Conclusion

This article reviews the application of NBCs in the remediation of heavy metal pollution and its synergistic mechanism. The results show that through in-situ doping and exogenous doping processes, the physical and chemical properties of biochar can be effectively regulated. The pore structure constructed by the hierarchical pyrolysis process and the abundant surface functional groups can enhance the adsorption performance of biochar. The analysis of chemical states such as pyridine nitrogen and thiophene sulfur reveals their important roles in the adsorption and catalytic processes. The regulation of the electronic structure by co-doping can further improve the interaction between biochar and heavy metal ions. Secondly, in terms of the remediation mechanism, the addition of synergistic oxidants activates the surface active sites of co-doped biochar, promoting the oxidative precipitation and chemical fixation of heavy metals. Nitrogen and sulfur can form stable complexes with heavy metal ions through complexation, significantly enhancing the ability to capture heavy metals. In addition, by optimizing raw material selection, preparation conditions, surface characteristic adsorption, and ion exchange adsorption, the remediation effect of NBCs can be further improved. Practical remediation cases show that NBCs has good application effects in the remediation of heavy metal-contaminated soil, providing support for the efficient and low-cost treatment of heavy metal pollution in the future.

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