CWAO Technology for the Treatment of Refractory-Biodegradable Organic Wastewater

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Abstract. With the development of society and industrial progress, the types and quantity of organic wastewater produced by refractory-biodegradation have increased. As an effective way to treat refractory-organic wastewater, CWAO technology has attracted more and more attention in recent years. The principle of CWAO technology belongs to free radical reaction, which mainly includes two processes: the exchange of oxygen in wastewater and the reaction between liquid oxygen and contaminants supported on the catalyst. Free radical reactions include three specific processes: chain initiation, chain delivery and chain termination. Homogeneous CWAO reaction can efficiently treat high concentration of organic wastewater, but the catalyst cannot be recovered to form secondary pollution. Heterogeneity is the research hotspot of CWAO technology and a suitable means for the treatment of highly concentrated organic wastewater, which has been used more and more in printing and dyeing, pharmaceutical, chemical, manufacturing and other industries.

1. Introduction

With the development of chemical industry and the progress of technology, the wastewater discharged by production has the characteristics of complex composition, high concentration and difficult to degrade, and has higher requirements for treatment technology. In recent years, the waste water discharge standards formulated by the Ministry of Environmental Protection have become more and more stringent, and the traditional physical, chemical and physicochemical treatment results have been unable to meet the corresponding national discharge standards. Therefore, the problem of treating the refractory-organic wastewater and discharging the standard needs to be solved.

Catalytic Wet Air Oxidation, abbreviated as CWAO, is an effective technology for the treatment of refractory-organic wastewater. Japan, the United States and European countries use CWAO to carry out a large number of experiments and apply it to industry. China has more and more research in this field, but it has not been applied to industrial scale treatment.

It can be found that in the process of treatment and degradation of flotation agents in mineral processing wastewater by catalyst [1], the methyl orange simulated printing and dyeing wastewater, landfill leachate and ceramic printing wastewater contain refractory-functional groups, which are more complex than the structure of mineral processing agent xanthate, so the refractory-organic compounds can be treated by catalytic wet oxidation. The impregnated catalyst is a new type of environment-
friendly catalyst, which is widely studied by experts and scholars at home and abroad. For its catalyst components, transition metals, rare earth metals and noble metals have been studied more [2]. Transition metals have the advantages of high COD removal rate and decolorization rate in degradation wastewater, but transition metals are easy to dissolve in acid wastewater and deactivate under alkaline conditions, resulting in poor stability or catalytic effect [3]. However, rare earth metals have special physicochemical properties that can inhibit component dissolution and improve catalytic activity, play a role of dispersant in catalyst, enhance the stability and resistance to sintering of catalyst, transform rare earth elements among different valence states in redox reaction, enhance the ability to transport oxygen, and then enhance the performance of catalyst to degrade wastewater and carry out more thorough reaction [4, 5]. The active components of impregnated catalyst are transition metal and precious metal. The auxiliary components include rare earth metal, which has no catalytic activity in itself, which can enhance oxygen transport and adjust the structure of catalyst, and promote the performance of transition-noble metal degradation wastewater. Transition metals as catalysts degrade wastewater with high activity but easy dissolution in wastewater, resulting in secondary pollution [6]; rare earth metals have stable properties and promote the transition metals to form stable solid solutions. On the one hand, the catalyst activity is improved, on the other hand, the oxides formed are not easy to dissolve and the stability is enhanced [7]; On the basis of rare earth-transition metal composite catalysts, doping noble metals metal, coupled with an efficient treatment process, so that the treated wastewater can meet the corresponding national discharge standards.

China is rich in transition metal and rare earth resources, easy to take materials, low price, and has a broad application prospect. High efficiency, stability and low price catalyst can contribute to the industrialization of CWAO technology to treat refractory wastewater. As a result, the preparation of high-efficiency catalyst, through CWAO treatment of refractory wastewater is the main research direction.

2. CWAO technology

2.1. Technical overview
The WAO concept was first studied and proposed by Zimmermann in the United States in 1944[8]. Since the 1970s, wet oxidation has also attracted the attention and research of environmental science in China. Cai [9] first applied WAO to petrochemical wastewater treatment in the early Ming Dynasty, and investigated the effect of reaction temperature, reaction pressure, influent concentration and process conditions on WAO process. Compared with the CWAO reaction with catalyst added in other literature, the experimental conditions were harsh, high cost and high demand for equipment, so the treatment of small amount and high concentration wastewater was good, but the large-scale treatment still needed to improve the WAO technology.

At the beginning of 1950, through the improvement of wet air oxidation method, the addition of catalyst in wet air oxidation method was widely studied. Through the role of catalyst, improve the treatment efficiency of organic wastewater degradation, reduce equipment investment, save operating costs and reduce the total cost.

2.2. CWAO radical reaction
The radical reaction is a complex process in the catalytic wet oxidation process, which consists of two main processes:

(1) Oxygen enters the liquid through the form of oxygen to complete the gas-liquid mass transfer exchange;

(2) The reaction between oxygen dissolved in the liquid phase and the catalyst loaded with pollutants.

Free radicals are usually divided into three stages: initiation, transfer and termination. The reaction process of free radicals is shown in Table 1.
2.3. Wet oxidation by homogeneous catalysis

The paragraph text follows on from the subsubsection heading but should not be in italic. When receiving the paper, we assume that the corresponding authors grant us the copyright to use the paper for the book or journal in question. Should authors use tables or figures from other Publications, they must ask the corresponding publishers to grant them the right to publish this material in their paper. The type of catalyst initially used in the CWAO method is homogeneous catalyst with good catalytic performance, low reaction temperature requirement and strong specificity.

In China, many scholars and experts have studied the properties of homogeneous catalytic wet oxidation organic wastewater. For example, Zhang et al. [10] used nitrate and sulfate of metals such as copper as the research object the COD removal rate and decolorization rate of methyl blue solution treated with degradation were higher than 71.1% and 84.7% at 120°C reaction temperature, 1.0 MPa oxygen pressure, respectively. Cu metal showed the highest catalytic efficiency, followed by Fe degradation performance, and the worst catalytic performance of Zr and Cr salts. Bai et al. [11] et tested at 220°C reaction temperature, 2.5 Mpa oxygen pressure. By comparing the nitrate catalyzed wet oxidized phenol wastewater from cu, zn and mn catalysts, cu had better catalytic effect than other metals, and its cod removal rate was as high as 97.2%; the catalytic effect of zn was the middle; and the catalytic performance of mn was the lowest. Tang et al. [12] studied the influence of medium factors of homogeneous catalysis on wet oxidation. Experiments show that the removal effect of wastewater can be greatly increased in the presence of catalyst. The COD removal rate of emulsion wastewater is ranked as Cu > Mn > Co, and oxygen content is also an important factor in catalytic wet oxidation. Fang et al. [13] used copper nitrate as catalyst and wet oxidation of ethoxyquinoline synthesis process wastewater. By investigating the effect of reaction temperature and catalyst dosage on the reaction, the experimental results show that increasing reaction temperature and catalyst dosage can increase the degradation rate of catalyst treatment wastewater. The COD removal rate of synthetic process wastewater was as high as 99.0% at 0.25 mg/ L dosing, 220°C reaction temperature and 8 MPa oxygen pressure.

There are also many experts and scholars abroad to study the homogeneous catalytic treatment of organic wastewater. Kurian et al. [14] used SOL-GEL method to prepare NiFe2O4 and ZnFe2O4 nanoparticle catalysts. The solution with degradation concentration of 1 g/ 25mL of P-Chlorophenol and the removal rate of target pollutants was 100% at 300 mg, reaction time of 60 min. Cihanoglu et al. [15] prepared Fe supported catalyst by ion exchange and treated acetic acid solution by fenton-like method. from the experimental results, the optimum conditions of the experiment were: 2.0 g/L dosage, reaction temperature of 333 K, hydrogen peroxide with pH concentration of 4 and 8.35, and acetic acid solution with concentration of 0.1 g/ dm³ under this condition, the COD removal rate was 50.5%.

The above domestic and foreign studies show that homogeneous catalysis shows excellent performance, but at the same time, it is easy to produce metal dissolution in homogeneous catalysis,

### Table 1. Free radical reaction process.

| No. | Process             | Mechanism of reaction                                                                 | Corresponding chemical equation                                      |
|-----|---------------------|---------------------------------------------------------------------------------------|---------------------------------------------------------------------|
| 1   | Initiation of chain | Organics produce HOO•, H₂O₂ and OH• active species in the presence of oxygen through contact with liquid oxygen | RH + O₂ → R• + HOO• (RH is organic) 2RH + O₂ → 2R• + H₂O₂ H₂O₂ + M → 2OH• (M is catalyst) |
| 2   | Transmission of chains | OH• radicals react further with organic matter, and the resulting R• reacts with oxygen to form ROO•, which interacts with organic matter | RH + OH• → R• + H₂O R• + O₂ → ROO• ROO• + RH → ROOH + R• |
| 3   | Chain stopping     | The whole reaction is terminated if the resulting R• interacts with or reacts with the ROO• itself and can not be contacted and reacted again by the free radicals | R• + R• → R─R ROO• + R → ROOR ROO• + ROO• → ROH + RCOR₂ + O₂ |
resulting in a large number of metal dissolution in organic wastewater to produce secondary pollution, which increases the subsequent cleaning process and increases the cost of the whole reaction, which is not conducive to the recycling of the catalyst.

2.4. Wet oxidation technology for heterogeneous catalysis

The stability of heterogeneous catalysts is better than that of homogeneous catalysts. Al₂O₃, CeO₂, TiO₂, SiO₂, activated carbon, CNTs and their combined substances are used as carriers to configure metal salts into solutions at a certain concentration. Heterogeneous catalysts are prepared by precipitation and impregnation methods [16] and applied to the treatment of organic wastewater. Heterogeneous catalysts exist in the form of solid state in the reaction, and the catalytic degradation of organic wastewater has good effect. It is easy to separate from organic wastewater after the reaction, and has strong recycling performance. The catalyst consists mainly of transition metals, rare earth metals and precious metals among them, transition metals have good catalytic activity, easy dissolution, and form homogeneous catalysis [17]; rare earth metals themselves have no catalytic activity, change the structure of the catalyst by doping, accelerate the contact of oxygen with other metals, produce more free radicals, and enhance the performance of wet oxidation of the catalyst [18]; noble metals have good catalytic effect and strong stability, but because the price is limited to large-scale applications.

2.5. Composition of catalyst components

Among heterogeneous catalysts, there is a need for a carrier first, which plays the role of carrying active components and catalytic auxiliaries. The carrier is porous and has a large specific surface area, mainly including Al₂O₃, activated carbon, molecular sieve, quartz sand and so on. In addition to the support, it can be divided into single component catalyst and composite component catalyst. The catalyst prepared by one metal salt is called single component catalyst, and the catalyst formed by mixing two or more metal salts is called composite component catalyst. Because of its singularity, the single component catalyst can not satisfy the increasingly complex degradation of refractory-organic matter, while the multi-component catalyst can utilize the unique advantages of various metals make up for the defects in the application of the catalyst, produce the synergistic effect, and produce better effect on the organic matter wastewater. According to the role in heterogeneous catalyst, the catalyst is divided into main catalyst and cocatalyst.

| No. | Ingredient | Key component |
|-----|------------|---------------|
| 1   | Carrier    | Al₂O₃, activated carbon, molecular sieve, quartz sand |
| 2   | Transition element | Cu, Fe, Co, Mn, Ni, Zr, etc. |
| 3   | Noble metal | Pt, Pd, Ru, Ti, Ir, etc. |
| 4   | Rare earth metal | La and Ce |

(1) Main catalyst

The main catalyst plays a major role in heterogeneous catalysts, is the main part of the catalyst, is an indispensable component. In the main catalyst group classification, there are both single component catalysts and composite component catalysts. Transition metals and noble metals are important components in the composition of main catalyst components.

Transition metals such as Cu, Fe, Co, Mn, Ni and Zr are used as catalyst components, showing excellent performance in the process of treating refractory-organic wastewater by catalytic wet oxidation, but in the process of use, the transition metal has poor stability, easy dissolution of metal components, and is difficult to recycle with the loss of organic wastewater, and cause secondary pollution to the wastewater, so it is generally combined with other metals to form a composite component catalyst. Ren et al. [19] prepared magnetic ferrite spinel MFe₂O₄ (M = Co, Cu, Mn, Zn) by
SOL-GEL method to investigate the performance of catalytic peroxy sulfuric acid-induced sulfate groups. The activity order was CoFe$_2$O$_4$ > CuFe$_2$O$_4$ > MnFe$_2$O$_4$ > ZnFe$_2$O$_4$, and the composite of Co and magnetic ferrite spinel showed excellent performance. Wijayapala et al. [20] used nickel nitrate as the nickel source and used the NiO/TiO$_2$ catalyst prepared by impregnation method to investigate the effect of different calcination temperature on the catalyst. The higher the temperature, the smaller the specific surface area of the catalyst, and the lower the activity of the catalyst. Chen et al. [21] used N doped C solid as the support, iron acetate as the main catalyst, and annealed at 800°C temperature. When the mass fraction of iron was 0.2%, the catalyst showed excellent conversion activity, stability and anti-toxicity, and pyridine nitrogen played a major role in the FeN$_4$ during catalytic oxidation. The Ni- Cu/ Al$_2$O$_3$- ZrO$_2$ nanocatalyst was prepared by impregnation method in rahemi et al. [22] to examine its catalytic performance of CO$_2$/CH$_4$ reforming, it was found that the activity of the catalyst in CO$_2$/CH$_4$ reforming did not decrease after the reaction temperature of 1,850°C , 1,440 min reaction, and showed good stability. Noble metals Pt, Pd, Ru, etc., as the main components of the catalyst, are often used to treat organic wastewater in the catalyst. Because of the expensive price of noble metals, in order to achieve a better catalytic effect and relatively low cost, the impregnation method is usually used to prepare the noble metal component catalyst, which not only makes full use of the efficient performance of the noble metal catalyst, but also reduces the amount of precious metal and enhances the recycling performance of the catalyst. Zhang et al. [23] prepared noble metal-modified core-shell structured M@TiO$_2$ (M= Au, Pd, Pt) catalysts supported by TiO$_2$. By degrading rhodamine B solution, the core-shell structured M@TiO$_2$ (M= Au, Pd, Pt) noble metal catalysts not only improved the activity of the catalysts to degrade wastewater, but also enhanced the recycling performance of the catalysts. Benkó et al. [24] used SiO$_2$ as support to prepare two-component metal catalysts with ag/au molar ratio of 10/90, 20/80, 33/67, 50/50 by sol adsorption. The catalyst with ag/au ratio of 33/67 showed good synergy in the oxidative decomposition of glucose. Mikulova et al. [25] used CeO$_2$, Zr$_{0.1}$(Ce$_{0.75}$Pr$_{0.25}$)$_{0.9}$O$_2$, Zr$_{0.1}$Ce$_{0.9}$O$_2$, and ZrO$_2$ as supports at 200°C reaction temperature, 2 MPa pure oxygen pressure to prepare catalysts by SOL-GEL method to study the catalytic performance of Pt on different supports for wet oxidation of acetic acid, and the mixed metal showed better oxygen storage, transportation and conversion ability, thus improving the catalytic performance of the catalyst, which made the conversion of acetic acid high.

(2) Catalyst

The catalyst itself has no catalytic activity, and the structure and performance of the catalyst can be changed effectively by a small amount of addition. According to its action, the catalyst can be divided into structure auxiliaries and electronic auxiliaries, which cannot change the structure of the catalyst, but can adjust the stability of the active components of the catalyst and increase the recycling performance of the catalyst. The electronic auxiliaries themselves have the ability to change the activity of the whole catalyst, which can promote the formation of active groups on the surface of the catalyst, thus increasing the effective collision between organic matter and catalyst, increasing the activity of the catalyst and increasing the stability of the catalyst.

Rare earth metals are often used as cocatalysts because of their special adjustment structure and catalytic properties. it not only shows special physical and chemical properties during the oxidation of organic matter, but also has a large ionic radius, which can form composite oxides such as solid solution that promote the formation of other metals. Commonly used rare earth metals include La and Ce. La is often used as a structural aid, while Ce is often used as an electronic aid. Ce rare earth metals are widely used in CWAO method, which is mainly due to the ability of Ce on the one hand to improve the dispersion of transition metals on the catalyst surface. On the other hand, due to the special redox cycle performance between Ce$^{3+}$ and Ce$^{4+}$, it can increase the oxygen storage capacity, promote the conversion of liquid oxygen to reactive oxygen species, improve the catalytic performance and enhance the stability of the catalyst. Li et al. [26] formed a Ce,Zr$_{1-x}$O$_2$ solid solution by Ce modification of Zr oxide, on which noble metal Ru was loaded. Treatment of O-chlorophenol by catalytic wet oxidation showed that the Ru/ Ce$_x$Zr$_{1-x}$O$_2$ catalyst with a mass fraction of 3% still maintained good catalytic performance at low temperature and low pressure. Arna et al. [27] prepared
the mnceox catalyst. The catalytic wet oxidation process for the treatment of phenol wastewater has found that the chemical components, calcination temperature, the addition of potassium metal and other factors seriously affect the performance of the catalytic wet oxidation of phenol. Zhao et al.[28] prepared TiO$_2$- CeO$_2$ and Fe/ TiO$_2$- CeO$_2$ catalysts by co-precipitation and impregnation, and treated 1-amino- 8- naphthol- 3, 6- disulfonic acid-sodium solution by catalytic wet-oxidation hydroxide. At 100$^\circ$C reaction temperature with initial ph of 5.0, the decolorization rate of 1-amino-8-naphthol-3,6-disulfonic acid-sodium solution was 98.1%, the removal rate of COD was 89.6% and the removal rate of TOC was 65.4%.

3. Conclusion
The treatment of refractory-organic wastewater is a difficult problem in the field of retarding pollution. Along with the progress of science and technology, CWAO technology has been paid more and more attention by researchers. CWAO technology can effectively treat refractory-organic wastewater, and its reaction includes two processes: oxygen exchange in wastewater and liquid oxygen reaction with pollutants. Radical reactions include chain initiation, chain transfer, and chain termination. The catalyst of homogeneous CWAO reaction can not be recovered to form secondary pollution, but the heterogeneous CWAO reaction can be recovered and reused after use. Heterogeneous CWAO technology in printing and dyeing, pharmaceutical, chemical, manufacturing and other industries get more and more applications.

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