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ARTICLE

PRESENT SITUATION AND PROSPECT OF CYANIDE CONTAINING ELECTROPLATING WASTEWATER TREATMENT PROCESS

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ABSTRACT

Discharge of electroplating cyanogen-containing Wastewater would cause great harm to the environment. The sources of cyanogen-containing wastewater were introduced, and several current domestic major physical and chemical processing technologies of cyanogen-containing wastewater were described and compared, which serve as a guide for engineering design. And future processing technologies of cyanogen-containing wastewater were discussed and prospected.

KEYWORDS

Cyanogen-Containing Wastewater, Hydrogen Peroxide Method, Biological Method, Clean Production Techniques

1. INTRODUCTION

Cyanide-containing wastewater is mainly produced in rare metal smelting and electroplating production. Among many kinds of plating, cyanide plating is one of the common plating types, mainly used for zinc, lead, cadmium, copper, silver, and gold plating. In cyanide-containing wastewater, in addition to highly toxic free cyanide, there are complex ions such as copper cyanide, cadmium cyanide, silver cyanide, and zinc cyanide. The concentration of CN- in wastewater is relatively high, and it also contains a large number of heavy metals, thiocyanate, and other compounds, which seriously pollutes the external water environment. Cyanide belongs to a highly toxic substance, and CN- will combine with a ferric cytochrome enzyme in the human body to generate ferric cytochrome oxidase and lose its oxygen transmission function, causing tissue hypoxia and suffocation in the body [1]. The lethal dose of cyanide in humans varies from person to person, from about 0.5 mg/kg to 3.5 mg/kg [2]. The lethal dose to other small animals (such as birds, etc.) and aquatic organisms are even smaller, which is a serious threat to the life safety of humans, animals, and aquatic organisms and destroys the ecological balance.

Although enterprises actively use a variety of different methods to treat cyanide-containing wastewater, many industrial and mining enterprises still discharge excessively. The paper introduces the recent development status of cyanide-containing wastewater treatment technology and briefly reviews the main treatment processes and technologies, which are conducive to giving inspiration to the innovation and improvement of cyanide-containing wastewater treatment technology.

2. BRIEF DESCRIPTION OF VARIOUS TREATMENT METHODS

There are many treatment methods for cyanide-containing wastewater in China [3,4], but which process is applied mainly depends on the mass concentration, properties, and actual treatment effect of cyanide-containing wastewater. The mass concentration of cyanide in wastewater can be roughly divided into three categories: high, medium, and low. Under normal circumstances, high-quality wastewater with complex composition CN > 800 mg/L. There are also various wastewaters with cyanide concentrations between (1-10) × 103 mg/L, so cyanide can be recovered by acidification first, and the residual liquid can be oxidized again. The medium concentration of cyanide-containing wastewater is generally between 200 mg/L and 800 mg/L. Therefore, the treatment process is selected according to the complexity of the wastewater components. If the wastewater components are simple and the recovery of cyanide is economical, it is suitable to first use the acidification method, and the residual liquid acquires the secondary treatment. The wastewater without economic benefit can be recovered by acidification and directly destroyed by oxidation. In actual domestic production, high and medium mass concentration (close to 800 mg/L) of cyanide-containing wastewater is generally determined by the process method according to the complexity of the components. In some simple wastewater, cyanide can be recovered first, and then the residual liquid can be directly oxidized to destroy CN-. The wastewater with medium and low mass concentrations adopts the direct oxidation treatment process. In recent years, there are many methods for recovering cyanide, such as acidification volatilization-alkali absorption method, extraction method, acid precipitation-neutralization method (two-step precipitation method), three-step precipitation method, etc. At present, few factories and mines use a single treatment process, because it is difficult for a single process to meet the national emission standards, so most of them use a combination of processes for treatment. The main combined treatment process is the technical combination of acidification recovery and direct oxidation, and the other combination is the technical combination of direct oxidation, natural purification [5], and the activated carbon adsorption process [6]. Many new wastewater full-cycle technology combination processes are also the main development of the trends. The selection of cyanide-containing wastewater treatment methods is mainly determined by the source, nature, and water volume of wastewater. These methods include the chemical method, physical-chemical method, physical method, and biochemical method, but the chemical method is the most used one. The following mainly introduces several commonly used physical and chemical methods for the treatment of cyanide-containing wastewater.

3. COMMONLY USED PROCESSING TECHNIQUES

3.1 Acid Aeration Method

This is a method that has entered the practical stage, and facilities of a certain size are being built in some countries such as the United States. Initially, the laboratory used aeration in neutral liquid to remove cyanide into the atmosphere. Later, it was improved to add acid to maximize the acidification of sewage, and then conduct aeration, which can remove cyanide more effectively. The acid used is usually sulfuric acid. Although there is also a proposal to use flue gas for acidification, it has not yet reached the mature stage, so it has not been popularized. The effect of this method is dominated by the degree of aeration and acidification. According to an example, when the pH is 2.8, aeration of sewage with a cyanide concentration of 500 mg/L can obtain a cyanide concentration of 0.09 mg/L. L-0.14 mg/L of treated water. Because after the implementation of this method, cyanide still maintains its original state and is discharged into the atmosphere as a toxic gas. It requires both favorable site conditions and a high chimney. Therefore, this method is only used in very limited areas. If liquid caustic is used to capture the vaporized cyanogen, it can not only make up for the above shortcomings but also recover the cyanogen.

3.2 Complex Salt Method

In the 1970s, some domestic enterprises ever used this method, but now they are not popular. From the point of view of environmental safety precautions, this method can be used as one of the quick remedial methods when cyanide produces a sudden pollution accident. The ferrous sulfate solution is put into water to quickly reduce the degree of harm caused by cyanide-containing pollutants in water and reduce harm to the environment, especially to aquatic organisms. When the concentration of CN- in wastewater is very low, the treatment effect of this method is not good. Although many kinds of medicines can be used, the most widely used is ferrous sulfate. This method utilizes ferrous sulfate to form a complex salt with cyanide, which is then precipitated and removed. The ferrous sulfate method converts cyanide to iron ferrocyanide, which is converted into a Prussian blue-type insoluble compound [7], which is then decanted or filtered.

$$FeSO_4 \bullet 7H_2O \longrightarrow Fe_2 + +SO_4^{2-} + 7H_2O$$

$$Fe^{2+} + 6CN^- \longrightarrow Fe(CN)_6^{4-}$$

$$2Fe^{2+} + Fe(CN)_6^{4-} \longrightarrow Fe_2[Fe(CN)_6]$$

It is characterized by simple operation, low treatment cost, and recyclable Prussian blue precipitate as a pigment. The disadvantage is that the treatment effect is poor, there is a lot of sludge, the wastewater after separation of insoluble is blue, and the concentration cannot be removed if the concentration exceeds a certain limit. From the perspective of the balance of the reaction, it is an unavoidable problem that the above-mentioned concentration is too high, and the removal rate decreases. Generally speaking, the pH value of the water is kept between 7.5 and 10.5 with lime, so that the formation of precipitation is at the highest level. However, even if the above measures are adopted, it will not be reduced because the cyanide content is below a certain value, and the effect is small when treating sewage with low cyanide concentration. If nickel is used as a treatment agent, although its effect is more favorable than iron, it is expensive. Xiong Zhengwei [8] conducted an experiment on the treatment of electroplating wastewater containing cyanide by the ferrous sulfate method and discussed the principle and removal effect of ferrous sulfate to remove the cyanide. The test results show that when the ferrous sulfate method is used to treat cyanide-containing wastewater from electroplating, the amount of ferrous sulfate added is 1.69 times the theoretical value, and when the amount of 0.1 %PAM flocculant is 1 mg/L, the removal rate of cyanide can reach 98%. It can also remove some heavy metal pollutants and COD, and COD can remove about 59%. The pH value has a great influence on the removal of cyanide. The generated ferrocyanide is converted into a relatively stable Prussian blue-type insoluble compound, and the cyanide removal effect is better when the pH value is controlled at 7.00-8.00.

3.3 Ozone Treatment Method

In recent years, research on the method of treating cyanide with ozone has been carried out quite widely, but due to the disadvantage of high electricity cost, it has not yet reached the general practical stage.

$$O_3 + KCN \longrightarrow KCNO + O_2$$

$$KCNO + O_3 + H_2O \longrightarrow KHCO_3 + N_2 + O_2$$

Ozone can release atomic oxygen to participate in the reaction in an aqueous solution, showing strong oxidizing properties, and can completely oxidize cyanide in a free state. Copper ions have a catalytic effect on the oxidative decomposition of cyanide ions and cyanide ions. Adding about 10 mg/L of copper sulfate can promote the decomposition reaction of cyanide. The outstanding features of the ozone method are that no other pollutants are added in the whole process, the amount of sludge is small, and the effluent is not easy to stink due to the increase of dissolved oxygen in the water. The ozone oxidation method is used to treat cyanide in wastewater, which only requires ozone-generating equipment and does not need to purchase and transport chemicals. The process is simple and convenient. The total cyanide mass concentration in the treated wastewater can reach the national comprehensive sewage discharge standard. There is no increase of other harmful substances in the waste liquid, no secondary pollution, and no need for further treatment. However, the industrial application is limited to a certain extent due to the high cost of ozone generated by the ozone generator and the difficulty of equipment maintenance. The industrial application prospect will be very broad as long as the ozone generator can break through the bottleneck of ozone generation. Ozone oxidation consumes a lot of electricity [9], and it is difficult to apply in places where electricity is lacking. Our country has already sold finished products of ozone generators, and some factories are currently using this treatment technology. It should be pointed out that the current ozone generator consumes a lot of energy, consuming 12 kW·h to 15 kW·h to produce 1 kg of O3, and the treatment cost is high. Except in a few places, it is generally difficult to meet the economic requirements of wastewater treatment. In addition, the use of ozone alone cannot completely oxidize the cyanide present in the complex state. Yan Haibo [10] used ozone technology to treat electroplating cyanide-containing wastewater. The concentration of CN- in electroplating cyanide-containing wastewater was between 30 mg/L and 36 mg/L. After treatment with activated carbon catalytic oxidation technology using ozone as an oxidant, the outlet concentration of CN- is less than 0.5 mg/L, and the removal rate is above 97.7%. The treatment system realizes the automation of wastewater treatment, and has the advantages of low investment, good effect, low cost, stable operation, etc., and will not produce secondary pollution, which is worthy of popularization and application.

3.4 Hydrogen Peroxide Method

3.4.1 Alkaline conditions

Under the conditions of normal temperature, alkaline (pH=9.5-11) and Cu2+ as a catalyst, H2O2 can oxidize free cyanide and its metal complexes (but not ferricyanide) into cyanate, and use metal cyanide metals such as copper, nickel, and zinc in the form of complexes, once the cyanide is oxidized

After the chemical is removed, they form hydroxide precipitates. The excess hydrogen peroxide also quickly decomposes into water and oxygen. Ferrocyanide in sewage is removed by copper precipitation. Its reaction equation is as follows. The equation for the reaction of free cyanide with hydrogen peroxide:

$$CN^- + H_2O_2 \longrightarrow CNO^- + H_2O$$

The equation for the reaction of metal complexes (excluding ferricyanide complexes) with hydrogen peroxide is as follows:

$$Me(CN)_4^{2-} + 4H_2O_2 + 2OH - \longrightarrow Me(OH)_2 \downarrow + 4CNO^- + 4H_2O$$

The reaction equation of ferrocyanide with copper is as follows:

$$Fe(CN)_6^{4-} + 2Cu^{2+} \rightarrow [Cu_2Fe(CN)_6] \downarrow$$

The excess hydrogen peroxide decomposition equation is as follows:

$$2H_2O \rightarrow H_2O + O_2$$

The cyanate salt generated in the above reaction is hydrolyzed to form ammonium ion and carbonate ion or bicarbonate ion, and the hydrolysis rate depends on the pH value. In general, thiocyanates are not or rarely oxidized. In the process of sewage treatment, the reaction sequence of cyanide-containing complexes is as follows:

$$\mathrm{Fe(CN)_6^{4-} > Zn(CN)_4^{2-} \quad [Cu(CN)_2]^- \quad CNS^-}$$

3.4.2 Acidic conditions

Generally, the wastewater is heated to 40 °C, and a mixed solution of H2O2 containing a small number of metal ions as a catalyst and 37% formaldehyde is added under constant stirring, and the reaction is completed by stirring for about 1 h. The reaction is carried out in two steps under acidic conditions:

First reaction:

$$CN^- + H_2O_2 \rightarrow CNO^- + H_2O$$

$$CN^- + HCHO + H_2O \rightarrow HOCH_2CN + OH^-$$

Secondary reaction:

$$HOCH_2CN + H_2O \rightarrow HOCH_2CONH_2$$

$$CHO^- + 2H_2O \rightarrow NH_4^+ + CO_3^{2-}$$

$$CN^- + 2H_2O \rightarrow HCOO^- + NH_3$$

This method is suitable for the treatment of cyanide-containing wastewater with large concentration fluctuations. There is no HCN gas generated in the whole process, and the operation is safe, but the required reagent costs are high. The Sanshandao Gold Mine of Shandong Gold Group Co., Ltd. uses hydrogen peroxide to treat the tail liquid after the acidification and recovery of cyanide-containing sewage [11]. The production and application situation in the past 1 year shows that this method has the advantages of simple process operation, low investment, and low cost, and can easily treat the acidification recovery tail liquid containing cyanide (CN)-5 mg/L-50 mg/L to a ＜0.5 mg/L, the drug cost is 7.56 yuan/m3.

3.5 Alkaline Chlorination Treatment

At present, the relatively mature technology for the treatment of cyanide-containing wastewater is to use the alkaline chlorination method. It must be noted that the cyanide-containing wastewater must be strictly separated from other wastewater to avoid mixing with metal ions such as nickel and iron, otherwise, the treatment will be difficult.

The possibility of decomposing cyanide by chlorine treatment has long been confirmed, but in the initial stage, chlorine treatment is carried out in an acidic solution, so a considerable concentration of toxic gas of hydrogen chloride is generated, and the operation is very unsafe. However, if chlorine treatment is carried out under alkaline conditions, the intermediate hydrogen chloride is converted to cyanate almost instantaneously, so this method has become a practical and safe method for cyanide treatment. The principle of this method is that the wastewater is destroyed and removed by chlorine-based oxidants under alkaline conditions. The treatment process is divided into two stages. The first stage is to oxidize cyanide to cyanate, which does not destroy cyanide., called the incomplete oxidation stage, the principle of this process is to oxidize cyanide to cyanate with hypochlorite under alkaline conditions (generally Ph ≥ 10).

$$CN^- + ClO - + H_2O \rightarrow CNCl\mathrm{+2OHCNCl+2OH^-} \rightarrow \mathrm{CNO^- + Cl + H_2O}$$

Combining the two equations, we get the following equation:

$$CN^- + ClO^- \rightarrow CNO^- + Cl^-$$

$$\square\square\square\square^{-} + 2 \quad {}_2 \rightarrow \quad {}_2 + \quad {}_3 + \quad {}^{-}$$

The toxicity of cyanate radicals generated by the cyanide breaking reaction by partial oxidation method is 1/1 000 of that of CN-, so in some factories, when the concentration of wastewater is relatively low, the wastewater is discharged into subsequent treatment facilities for metal ions after partial cyanide breaking treatment. However, CNO- is a toxic substance after all, and is easily hydrolyzed to ammonia (NH) 3 under acidic conditions. pH reaction condition control: primary oxidation to break cyanide: value 0-11; theoretical dosage: simple cyanide CN-: Cl2=1:2.73, complex cyanide CN-: Cl2=1:3.42. The ORP instrument was used to control the reaction endpoint to be 300 mv-350 mv, and the reaction time was 10 min-15 min.

The second stage is to further oxidize and decompose the cyanate into carbon dioxide and water, which is called the complete oxidation stage. Based on partial oxidation treatment, the pH of wastewater is adjusted (generally pH≥8.5), and then a certain amount of oxidant is added, and CNO- is completely oxidized to N2 and CO2 by stirring.

$$2CNO^- + 3ClO^- \rightarrow CO_2 \uparrow + N_2 \uparrow + 3Cl^- + CO_3^{2-}$$

The pH reaction condition control: secondary oxidation to break cyanide: pH value 7-8 (adjusted with H2SO4); theoretical dosage: simple cyanide CN-: Cl2=1:4.09, complex cyanide CN-: Cl2=1:4.09. Use the ORP instrument to control the endpoint of the reaction to be 600 mv-700 mv; the reaction time is 10min-30min. The residual chlorine concentration in the reaction effluent is controlled at 3 mg/L-5 mg/L. Teng Huamei [12] and others used a two-stage alkaline chlorination treatment process to treat the cyanide-containing wastewater from Hangzhou Xierlingzhong Factory. The intermittent method was used to control the dosage manually. The concentration of cyanide in the original wastewater was 59.8 mg/L-141.1 mg/L. L, the average is 84.6 mg/L, the pH is adjusted in sections, stirred with a self-made mechanical stirrer, and the dosage is calculated in sections according to the cyanide concentration measured in the laboratory. The wastewater treatment has achieved good results. The cyanide concentration is less than the national emission standard of 0.5mg/L. In addition, sodium hypochlorite, sodium chlorite, bleaching powder, etc. are used to replace chlorine gas. The principle and method are the same as those of chlorine gas, but special devices similar to chlorinators are no longer required, and the danger of chlorine gas leakage can be avoided. It is suitable for small-scale wastewater treatment. Where such a treatment method has been decided, the effect of residual chlorine at the discharge destination must be considered.

3.6 Salt Electrolysis Method

Chlorine gas and strong alkali are simultaneously generated by the electrolysis of salt water, and they are used for the decomposition of cyanogen. As far as electroplating plants are concerned, it is easy to operate because of easy access to electricity supply, and the cost of handling chemicals is very low. Especially in the case of batch operation, the rectifier originally used for the plating operation can be fully utilized in idle time at night, so the equipment cost can also be reduced. The disadvantage of this method is the short service life of the carbon electrode used for the electrolytic anode. It is suitable for smaller-scale factories.

(1) Diaphragm electrolysis method: This is a method of using a diaphragm in the salt electrolysis method, and its principle is an alkaline chlorination treatment method. If there are many impurities in the salt, the asbestos used in the diaphragm is prone to the disadvantage of clogging the gap. In the case of continuous operation, if the management is not good, it is easy to cause insufficient salt supplementation, so the decomposition reaction cannot continue. Therefore, frequent attention must be paid.

(2) Non-diaphragm electrolysis method: During non-diaphragm electrolysis of salt water, chlorine gas is generated on the anode, and it reacts with the alkali generated on the cathode to generate hypochlorite.

$$Cl_2 + 2NaOH \rightarrow NaOCl - NaCl + H_2O$$

If the generated chlorate is added to the sewage containing cyanide, the cyanide will be oxidized to form cyanate.

$$NaCN - NaOCl \rightarrow NaCNO + NaCl$$

And the cyanate will be further decomposed into carbon dioxide and nitrogen.

$$2NaCNO + 3NaOCl + H_2O \rightarrow 2CO_2 + N_2 + NaOH + 3NaCl$$

4. APPLICATION PROGRESS OF BIOLOGICAL TREATMENT METHODS FOR CYANIDE-CONTAINING WASTEWATER

Some scholars [13] used the BOD5/COD ratio method and the aerobic respiration curve method to conduct a comprehensive aerobic biodegradability study on high-concentration organic cyanide wastewater and its pollutants for the first time at home and abroad. The results show that the biodegradability of cyanide-containing wastewater is good at low concentrations and poor at high concentrations. Even high concentrations cannot be biodegraded by aerobics. Xiao Min [14] et al. measured the anaerobic biodegradability of acrylonitrile, acrylic fiber production process wastewater, and other highly concentrated organic cyanogen wastewater and the toxicity of acrylonitrile, acetonitrile, cyanide, and other major pollutants in wastewater to methanogenic bacteria by using serum bottle liquid replacement system and air anaerobic hydration reaction equipment conditions at 30 °C. The results showed that acrylonitrile was a metabolic toxin at low mass concentration, the methanogenesis activity of anaerobic bacteria was recovered in the recovery test. It was a physiological toxin at a high mass concentration (>120 mg/L), and the methanogenic activity caused by toxicity was inhibited but recovered in a short period. Cyanide is physiologically toxic at low mass concentrations and bactericidal toxins at higher mass concentrations (25 mg/L) in which anaerobic cells have been severely damaged and cannot be repaired. Acetonitrile is always a metabolic toxin. Zhang et al. [15] used membrane separation technology to treat acrylonitrile-containing cyanide wastewater. After treatment, the cyanide ion concentration CN-<0.0005 % and COD<1 500 mg/L, which showed that the use of an ultra-filter membrane can effectively purify the raw water and reduce the COD content of the raw water to a certain extent.

5. CONCLUSION AND OUTLOOK

The above discussion shows that there are many treatment methods for cyanide-containing wastewater, and the characteristics of the methods are different. Due to the differences in the source, concentration, treatment purpose, scale, and economic requirements of the wastewater, these methods have their advantages and disadvantages. Since cyanide does not appear alone in sewage, it is often accompanied by other inclusions, so even if any treatment method has been decided, it is still necessary to consider the treatment of other inclusions. Therefore, it is impossible to determine which method is the best for the removal of cyanide alone, or how large it should be designed. In the actual design, other conditions must be taken into account to formulate a plan for removing cyanide. In particular, the oxidative destruction technology of cyanide-containing wastewater and the cyanide total recycling technology has been relatively mature in industrial production, but various processes are not perfect and need to be further improved. The current electroplating cyanide-containing wastewater has entered the stage of comprehensive prevention, recycling, and total control. The treatment of cyanide-containing wastewater should start from the root cause, adopt comprehensive prevention and control technology to avoid pollution, and start with cleaner production technology, supporting some comprehensive utilization. Practical processing technology makes the treatment effect more perfect. Therefore, in the future, the treatment of electroplating wastewater containing cyanide will highlight the following aspects:

(1) The focus of environmental protection management is shifted from the end to the source, starting from the control of raw materials, implementing whole-process control, reducing the number of pollutants, and adopting comprehensive treatment of wastewater to achieve the lowest pollution discharge. "Wastewater reuse system devices", easy to operate and low in operation cost, will be developed to maximize the recycling of water.

(2) With the application of genetic engineering, molecular engineering, molecular biology, and other technologies, biotechnology has shown great development potential including the advantages of low cost, high benefit, and no secondary pollution. We should make full use of the synergistic purification effect of natural microorganisms and plants, supplemented by physical or chemical methods, to find an effective way to purify pollutants, which has practical significance for the treatment of cyanide-containing wastewater.

(3) Actively researching the recovery and utilization of cyanide in cyanide-containing wastewater is of great significance for saving social resources.

(4) Further research on non-pollution treatment technology and advanced treatment technology should be carried out to reduce the treatment cost in terms of the reuse of pollutants and the reuse of treated water.

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