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Application of modified bentonite granulated electrodes for advanced treatment of pulp and paper mill wastewater in three-dimensional electrode system

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ABSTRACT

Two types of granular electrodes (cetyl trimethyl ammonium bromide modified bentonite (CTAB-bent) and hydroxy-aluminum pillared organic bentonite (OH-Al-CTAB-bent)) were prepared and form a three-dimensional (3-D) electrode system with general electrode plates to dispose the pulp and paper secondary wastewater deeply of paper mills. Then XRD, FTIR and SEM spectroscopy analysis were applied to characterize the feature of the electrodes. The removal efficiency of the chemical oxygen demand (COD) and color of the effluent after treatment was investigated. It was found that the removal efficiency of COD and color depended on the reaction time, current density, amount of granular electrode, the airflow and the run times. The results proved that the 3-D electrode system exhibited high efficiency in the removal of COD and color. Under the optimum conditions, the COD could be degraded from initial 256 mg/L to 40 mg/L, that was, 84.3% of COD were removed. And the removal efficiency of color could reach 93%. So the secondary wastewater treated advanced could meet the discharge standard (GB3544-2008). Finally, the possible reaction mechanism of organic pollutants degradation in 3-D electrode system was expatiated.

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1. Introduction

Pulp and paper industry is one of the largest water polluters in the world. The high water usage results in large amounts of wastewater generation [1]. It has been estimated that the pulp and paper industry is responsible for 50% of all wastes dumped into Canada's waters [2]. Recent years, for example in China, the discharge of national industrial wastewater increased year by year and the emission loading of COD and NH_3-N was also growing [3]. The various papermaking processes and abundant addition of chemicals lead the wastewater to be complicated and contain a heavy burden of dissolved and semisoluble or undissolved organics, usually measured as chemical oxygen demand (COD), biochemical oxygen demand (BOD), suspended solids (SS), absorbable organic halides (AOX) and colorants, etc., which can cause many serious environmental problems [1]. Generally, the organic pollutants in wastewater can be classified into five categories (Table 1) [4]. In raw sewage, these organic compounds correspond to COD of 2 or 3 g/L [5].

Commonly, pulp and paper wastewater all need secondary treatment. The primary treatment aims to remove the pollutants such as suspended solid (SS), grease, and colloid through mechanical and physical treatment. However, the dissolved pollutants were still retained in the wastewater after this step. So the secondary treatment (biochemical treatment) becomes very important. The pollutants can be degraded and convert to sludge due to the action of microorganisms. A review of the results for pulp mills in Finland shows that such techniques remove 73–99% of BOD and 50–92% of COD [6].

However, the wastewater is still hard to reach the discharge standards after secondary treatment. Many toxic compounds in wastewater such as lignin and its derivatives are very difficult to be degraded [7]. The effluents still contain various biorefractory organic matters [8]. Furthermore, the aroused pollution incidents have attracted the attention of the public and the governments. Many studies have been focus on the problem as well as the control of the pollutants [9]. In China, the new standard gives stricter emission standard (GB3544-2008: pH: 6–9; color (dilution times): 50; COD_{Cr}: 50 mg/L) compared to GB3544-2001. Because of water shortage, pulp and paper industries must save freshwater, reduce

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Table 1
Organic pollutants in paper wastewater.

Categories	Organic pollutants
Carbohydrates	Glucose, galactose, etc.
Middle extractives	Resin and fatty acids, etc.
Lignans	Lignin polymers, benzene series, etc.
Lignin and its phenolic derivatives	Aromatic alcohol, etc.
Low molecular weight compounds	Alcohol, alkane, etc.

Table 2
Physical properties of Na-bent.

Physical properties	Na-bent
Distensibility capacity (mL/g)	14
Colloid index (mL/g)	21
Volume traded orchid (g/100 g)	40.2
CEC (mmol/100 g)	87.4
Conductivity (10^{-3} S cm^{-1})	0.3

the production of wastewater and improve the recycling rate of clean and treated water. Hence, it is necessary to develop a tertiary treatment to improve wastewater discharge quality and to allow the inclusion of wastewater as process water [5,8].

Electrochemical oxidation is attracting more and more attention because of its simple equipment, high efficiency, and low solid wastes created and so on. In particular, electrochemical technologies based on three-dimensional electrode have been attracting much more attention [10–13]. Comparing with conventional two-dimensional electrode, three-dimensional electrode is equipped with larger specific surface area, higher performance. Therefore they have a broad and interest application prospect [14,15]. In the past decades, the three-dimensional electrodes have been successfully used to handle various industrial wastewaters containing petroleum refinery wastes, dye wastes, phenolic resin wastes, phenol waste and heavy metal-containing solutions [10–14].

The type of three-dimensional electrode has experienced various kind of evolution since it was proposed in 1960s [10]. In the initial days, bed electrodes such as packed or fluidized bed electrode were used frequently, granular electrode of different sizes such as activated carbon (GAC) and graphite are the main materials in forming the bed and acted as three-dimensional electrode [14–16]. Later, integral type electrode was exploited, some new materials such as reticulated vitreous carbon, titanium foams were used by researchers [17,18]. Recently, the application of novel type of electrode particle has attracted more and more attentions. Granular electrode is a new style particle electrode and this kind of particle is multifunctional due to its unusual characteristics, such as high specific surface area, perfect chemical stability, and excellent conductivity, and it is suitable to use as three-dimensional electrode [19,20].

There are some advantages for granular electrode than the porous electrodes or integral type electrodes. At first, it can overcome the inherent disadvantages of uneven potential distribution created due to the structure of the granular electrodes, and overcome the current distribution and effective of electrochemical oxidation in turn [21]. Secondly, since the specific surface area of granular materials is much bigger than the rest type of electrodes and it is facile to combine with the reaction media. Moreover, the specific surface area of the electrode is directly proportional to the rate of electrochemical reaction and electron transfer [10]. Finally, granular electrodes also have perfect adsorbent ability, this provides more opportunities for the adsorption of the organic compounds in the wastewater, and provides more opportunities for the happening of electro-catalytic oxidation process, thus enhance the decompose efficiency of the organic pollutants [19].

However, there are few literatures about the exploitation of new granular electrode materials [19,20]. Nowadays, there is a remarkable reemergence of interest in the preparation of pillared layered materials, such as bentonite is one of the most widely used abundant and low-cost natural clay minerals [22]. In the past researches, it is usually used as adsorbent after organic modification. The characteristics such as adsorption capacity, conductivity can increase obviously after organic modification because of the surface properties of bentonite are transformed from hydrophilic to hydrophobic and oleophilic [23–28]. And it was proved that some organic modified bentonites can adsorb organics pollutants effectively which have the similar chemical characteristics in pulp and paper water, such as benzene series, esters, phenol, tannin and its chlorinated compounds [27,28]. It is worth noting that the stability of the organic clays mineral or soil synthesized by cationic surface active agent and clay minerals or soil is related to the reaction form. The organic clay or soil produced through ion exchange is more stable and difficult to be decomposed. Even under the condition of long time, extreme pH, high ionic strength medium and organic solvent, the organic clay or soil is still difficult to desorb [29,30]. So the researches about the application of organic modified bentonite in synthesis of other materials are the latest.

In this study we synthesized two type new modified bentonites as the third electrode in the 3-D system to deal with secondary treatment wastewater of pulp and paper mill. The impact of electrolysis time, current density, the dosage of modified bentonite and electrode material on COD removal efficiency and color removal efficiency were also explored. Finally, the optimum parameters of this study were determined. In addition the possible mechanism of the degradation of organic pollutants in the 3-D electrode system was proposed.

2. Materials and methods

2.1. Materials

Sodium bentonite (Na-bent) was from Xinyang xiongshi Co., Ltd., Henan province, Table 2 shows its physical properties. The CTAB and other chemicals all were class AR.

The target secondary wastewater was from Shandong Huatai paper industry, China. Table 3 shows its characteristics.

2.2. Preparation of granular electrodes

2.2.1. Synthesis of CTAB-Bent

The Na-bent (10 g) and deionized water (180 mL) was mixed in 500 mL three round-bottom flask and stirred for 30 min. Then the modifier agent CTAB was dispersed into the suspension and the mixture suspension was stirred for 2 h. When the reaction was finished, the product was washed repeatedly by deionized water until Br^- was wiped off. The product should to be dried at 85 °C. After that the dry bentonite must be made to be granular and activated for 2 h at 105 °C. The end product CTAB-bent needed to be saved in desiccator and calculate the yields.

2.2.2. Synthesis of OH-Al-CTAB-Bent

The synthetic method accorded to the relative contents described by Zeng xiuqiong [31]. Adding NaOH (0.5 M) slowly into a 500 mL three round-bottom flask containing $AlCl_3$ (60 mL, 1 M) and stirred the mixed solution constantly for 3 h at 70 °C. Then the pillaring solution was stored for 24 h at 60 °C.

The OH-Al-CTAB-bent was synthesized by adding pillaring solutions to a CTAB-bent suspension drop-wise and stirring for 2 h at 80 °C and storing for 48 h at 60 °C. The product was washed several times with deionized water until there was no chloride ion.

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