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Removal of total cyanide in coking wastewater during a coagulation process: Significance of organic polymers

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ABSTRACT

Whether a cationic organic polymer can remove more total cyanide (TCN) than a non-ionic organic polymer during the same flocculation system has not been reported previously. In this study, the effects of organic polymers with different charge density on the removal mechanisms of TCN in coking wastewater are investigated by polyferric sulfate (PFS) with a cationic organic polymer (PFS-C) or a non-ionic polymer (PFS-N). The coagulation experiments results show that residual concentrations of TCN ($\text{Fe}(\text{CN})_6^{3-}$) after PFS-C flocculation (TCN < 0.2 mg/L) are much lower than that after PFS-N precipitation. This can be attributed to the different TCN removal mechanisms of the individual organic polymers. To investigate the roles of organic polymers, physical and structural characteristics of the flocs are analyzed by FT-IR, XPS, TEM and XRD. Owing to the presence of N^+ in PFS-C, $\text{Fe}(\text{CN})_6^{3-}$ and negative flocs ($\text{Fe}(\text{CN})_6^{3-}$ adsorbed on ferric hydroxides) can be removed via charge neutralization and electrostatic patch flocculation by the cationic organic polymer. However, non-ionic N in PFS-N barely reacts with cyanides through sweeping or bridging, which indicates that the non-ionic polymer has little influence on TCN removal.

Introduction

Cyanides are chemical compounds which contain a cyano group ($\text{C}\equiv\text{N}$), consisting of a carbon atom triple-bonded to a nitrogen atom. Many kinds of cyanide compounds are highly toxic because they release the cyanide ion (CN^-) (Dasha et al., 2009; Patil and Paknikar, 2000). To reduce the concentration of the cyanides in wastewater, an alkaline chlorination process has been widely used. This process destroys free dissolved hydrogen cyanide and oxidizes all simple and some complex inorganic cyanide in aqueous media (Young and Jordan, 1995). However, when the

cyanides exist in the form of complexes, such as ferrocyanides or ferricyanides, the alkaline chlorination process has difficulty treating them (Parga et al., 2003). Thus, a variety of alternative removal technologies (Barakat et al., 2004; Kitisa et al., 2005; Barriga-Ordonez et al., 2006; Shen et al., 2006; Park et al., 2008; Wiggins-Camach and Stevenson, 2011) have been developed, including ozone oxidation, hydrogen peroxide oxidation, electrochemical oxidation, chemical precipitation, photocatalysis, and coagulation-gas-filled membrane separation. These technologies theoretically could remove cyanides with high efficiency, however their application is limited due to treatment cost and special equipment requirements.

Coagulation-precipitation is one of the most common technologies in wastewater advanced treatment (Sharp et al., 2006). For example, during the coking wastewater

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treatment process, inorganic coagulants and a non-ionic organic polymer (polyacrylamide), are usually applied to advanced treatment to further remove residual chemical oxygen demand (COD), turbidity, and color (Yang et al., 2011). However, the final effluents still contain a certain concentration of total cyanide (TCN: 4–5 mg/L), which is still beyond the regulation level of China (TCN < 0.2 mg/L) (GB 16171-2012).

Previous investigation (Park et al., 2008) demonstrated that chemical precipitation methods, such as Prussian blue formation, were used to treat cyanide-containing effluent from biological coking wastewater. Furthermore, organic polymers have been used as flocculants to improve cyanide removal efficiency. For example, Kleefish (1992) used a synthetic cationic polymer with ferric sulfate for precipitation of TCN from coke oven wash waters. During the coagulation process, the organic flocculants agglomerated and flocculated the ferricyanides, and cyanide removal rate was 80% or higher. Yablonsky (1995) also used a cationic polyelectrolyte to TCN removal efficiency.

Previous researches indicated that cationic and non-ionic organic polymers showed significant differences in TCN removal efficiencies. However, whether a cationic organic polymer has superior efficiency in TCN removal when compared to a non-ionic organic polymer during the same flocculation process which has not been reported previously. In particular, the difference in TCN removal mechanisms still needs to be further investigated.

The primary objectives of the present research were to determine the mechanisms of TCN removal by different flocculants including cationic and non-ionic polymers. The experiments were conducted during coagulation/flocculation of coking wastewater effluent after biological treatment (CBW). To make clear TCN removal mechanisms, the TCN removal efficiencies, floc physical properties and variation of floc surface functional groups were investigated. The physical properties of flocs were examined using zeta potential and floc growth dynamics. Transmission electron microscopy (TEM) and X-ray diffraction (XRD) were used to observe the morphology and crystallinity of flocs. Moreover, Fourier transform infrared spectroscopy (FT-IR) and X-ray photoelectron spectroscopy (XPS) were used to analyze the migration of surface functional groups on flocs.

1 Materials and methods

1.1 Preparation of coagulants

Inorganic pre-polymerized polyferric sulfate (PFS) ($r = [\text{OH}]/[\text{Fe}]$, i.e., 0.25; the total concentration of Fe was approximately 25.0% \pm 5.0% (W/W) in the PFS) was purchased from Shandong Dongjia Group Co., Ltd., Zibo, China. Cationic (KL-107-C) and non-ionic polymers (KL-

110-N) were obtained from Saike Kanglun Environmental Protection and Technology Co. Ltd., Beijing, China (for detailed information, see **Table S1**). Coagulants were prepared by mixing PFS and organic polymers (KL-107-C or KL-110-N) and were labeled PFS-C and PFS-N, respectively. To prepare the coagulants, a measured amount of organic polymer was injected into the PFS solution at a fixed rate while stirring thoroughly until it was absolutely mixed. To prevent the formation of insoluble by-products, the preparation process was carried out at 50°C. The properties of the coagulants are listed in **Table 1**.

1.2 Coagulation experiments

The coagulation experiments were carried out in the coagulation-flocculation operation units of a wastewater treatment system (for a schematic diagram, see **Fig. S1**). The wastewater to be treated was biological effluent with a flow rate of 150–200 m³/hr. The properties of the wastewater are presented in **Table S2**. To compare coagulation efficiencies with different coagulants, the coagulation process was divided into two systems (1[#] and 2[#]). PFS-C and PFS-N were injected into systems 1[#] and 2[#], respectively. In this investigation, the flow rate of each system was maintained at 75 m³/hr. The dosages were adjusted every three days. Furthermore, to evaluate the coagulation stability of the coagulants, the optimal dosages were maintained for eighteen days. The samples were collected at a fixed time every day, and sampling locations are presented in **Fig. S1**.

1.3 Analysis of wastewater parameters

COD_{Cr} was measured by a closed reflux spectrophotometric method on a UV-Vis spectrophotometer (UV9100A, Labtech Co. Ltd., USA). The standard methods were employed for measurement of TCN concentration (GB 7486-87). The zeta potential of colloidal particles was measured by a Beckman Coulter DelsaNano analyzer (USA).

1.4 Characterization of flocs

The flocs were dried by freeze-drying at –70°C for 96 hr. Selected floc samples were characterized by XRD for the determination of crystalline phases using a multifunctional X'Pert PRO MPD XRD (PANalytical Company, Holland) with CuK radiation in the 2 θ range of 10–90° at a scan rate of 10°/min. The morphology of the flocs was studied by

Table 1 Properties of coagulants

Property	PFS-C	PFS-N
Density (g/L)	1.15	1.21
pH	1.20 \pm 0.05	1.18 \pm 0.08
Color	Reddish brown (liquid)	Reddish brown (liquid)
Weight percents (W/W)	10%	10%

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