

Inhibitory effects of toxic compounds on nitrification process for cokes wastewater treatment

Young Mo Kim^a, Donghee Park^a, Dae Sung Lee^{b,*}, Jong Moon Park^{a,**}

^a Advanced Environmental Biotechnology Research Center, School of Environmental Science and Engineering/Department of Chemical Engineering, Pohang University of Science and Technology, Pohang 790-784, South Korea

^b Department of Environmental Engineering, Kyungpook National University, Daegu 702-701, South Korea

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Abstract

Cokes wastewater is one of the most toxic industrial effluents since it contains high concentrations of toxic compounds such as phenols, cyanides and thiocyanate. Although activated sludge process has been adapted to treat this wastewater, nitrification process has been occasionally upset by serious inhibitory effects of toxic compounds. In this study, therefore, we examined inhibitory effects of ammonia, thiocyanate, free cyanide, ferric cyanide, phenol and *p*-cresol on nitrification in an activated sludge system, and then correlated their threshold concentrations with the full-scale pre-denitrification process for treating cokes wastewater. Ammonia below 350 mg/L did not cause substrate inhibition for nitrifying bacteria. Thiocyanate above 200 mg/L seemed to inhibit nitrification, but it was due to the increased loading of ammonia produced from its biodegradation. Free cyanide above 0.2 mg/L seriously inhibited nitrification, but ferric cyanide below 100 mg/L did not. Phenol and *p*-cresol significantly inhibited nitrification above 200 mg/L and 100 mg/L, respectively. Meantime, activated carbon was added to reduce inhibitory effects of phenol and free cyanide.

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1. Introduction

Cokes wastewater is generated in the coal coking, coal gas purification and by-product recovery processes of cokes factory in iron and steel industries. The chemical composition of cokes wastewater is very complex and varies from one factory to another, depending on the quality of raw coal, carbonation temperature and method used for by-products recovery [1]. Phenolics are the main organic constituents, accounting for about 80% of the total COD. Other organics include polynuclear aromatic hydrocarbons (PAHs) and heterocyclic compounds containing nitrogen, oxygen and sulfur [1,2]. Inorganic constituents are mainly composed of cyanides, thiocyanate and ammonia [1,3–5]. Thus, cokes wastewater has been considered the most toxic one to be treated before being discharged into the environments [1–3].

Traditional treatment of high-strength cokes wastewater utilizes expensive caustic treatment and steam stripping to reduce the contaminant load, followed by conventional biological treatment [6–9]. Among various proposed processes [1–9], a biological nitrogen removal process, especially pre-denitrification process, has been preferred in Korea because of its simplicity and economic benefits [3]. The pre-denitrification process is a single-sludge system with recycle of nitrified effluent and consists of two distinct microbial reactions under anoxic followed by aerobic conditions (Fig. 1). These successive microbial reactions remove total nitrogen within the wastewater [3]. Meanwhile, toxic compounds such as phenols, cyanides and thiocyanate are degraded by various aerobic or anaerobic microorganisms present in the activated sludge system.

However, a full-scale pre-denitrification process for treating cokes wastewater has occasionally failed to remove total nitrogen below its discharge level (50 mg/L). Kim et al. reported that fast growth of competitive microorganisms at higher temperature might cause washing out of nitrifying bacteria and resulting in instability of nitrification process during summer [3]. However, there is less information on the reasons of abrupt failure

* Corresponding author. Tel.: +82 53 950 7284; fax: +82 53 950 6579.

** Corresponding author. Tel.: +82 54 279 2275; fax: +82 54 279 2699.

E-mail addresses: daesung@knu.ac.kr (D.S. Lee), jmpark@postech.ac.kr (J.M. Park).

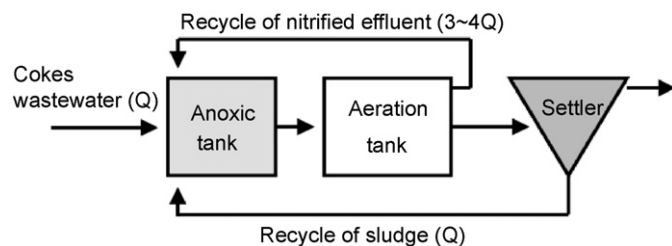


Fig. 1. Schematic diagram of a full-scale pre-denitrification process.

in the full-scale process regardless of the season. It might be due to incomplete nitrification by inhibitory effects of pollutants present in the cokes wastewater. Although many researchers have reported the inhibitory effects of toxic compounds such as phenol and free cyanide on nitrifying bacteria [10–14], there is less information on their threshold concentrations causing inhibitory effects on the full-scale process. In addition, there is less or no information on reliable guidelines for preventing these inhibitory effects.

In this study, we examined inhibitory effects of ammonia, thiocyanate, free cyanide, ferric cyanide, phenol and *p*-cresol on nitrification in an activated sludge system, and then correlated their threshold concentrations with the full-scale pre-denitrification process for treating cokes wastewater by considering biochemical reactions and full-scale operational conditions such as dilution ratio. Meantime, the effect of activated carbon addition for reducing inhibitory effects of toxic compounds was examined.

2. Materials and methods

2.1. Activated sludge and wastewater

Activated sludge used in this study was collected from an aeration tank of a full-scale wastewater treatment facility of a cokes manufacturing plant in a steel company, Korea. It was very difficult to collect sludge-free wastewater flowing into the aeration tank where nitrification reaction occurs. As an alternative, test solutions were prepared by dissolving the exact quantities of phenol (Junsei), *p*-cresol (Acros), KCN (Acros), $K_3Fe(CN)_6$ (Kanto), KSCN (Junsei) and NH_4Cl (Samjun) in sludge-free effluent obtained from a settler of the wastewater treatment process (Note that only these toxic compounds were absent in the effluent, as shown in Table 1). Additionally, 1 g/L of $NaHCO_3$

(Samjun) was added in the test solution to supply inorganic carbon for nitrifying bacteria.

2.2. Batch experiments

Batch experiments for nitrification reaction were carried out in 500 mL Erlenmeyer flasks filled with 200 mL of test solution containing 50 mg-N/L of ammonia and other toxic compounds. Each flask was inoculated with activated sludge in the range of 2640–2800 mg/L, and then agitated on a thermostatic shaker at 200 rpm and 30 °C. During batch experiments, dissolved oxygen concentration was above 3 mg/L, and the solution pH was continuously maintained at 7.5 using 1 M NaOH or 1 M HCl solution. 1 mL of samples was intermittently removed from the flask to analyze ammonia, nitrate, nitrite, phenol and thiocyanate concentrations. It was confirmed from three independent replicates that the nitrification experiments were reproducible within at most 5% error in this study.

2.3. Analytical methods

Each sample collected from batch experiments was centrifuged at 3500 rpm for 3 min with a centrifuge (MF550, HANIL), and then the supernatant was used for the following analyses. Ammonia was analyzed with phenate method, phenol with chloroform extraction method and thiocyanate with reaction with ferric nitrate using a spectrophotometer (GENESYS TM 5, Spectronic Inc.) [15]. Total cyanide concentration was determined by pyridine-pyrazolone method after distillation. To measure free cyanide concentration, free cyanide was tripped by vigorous air-blowing and absorbed into a 1 M NaOH solution, and then the solution was used for cyanide analysis. Nitrite and nitrate concentrations were measured with an ion chromatograph (DX-120, DIONEX Co.).

3. Results and discussion

3.1. Effect of ammonia on nitrification

Although nitrifying bacteria use ammonia as an electron source for energy, nitrification reaction can be inhibited by ammonia itself at high concentration [16]. Fig. 2 shows the effect of initial ammonia concentration (50–500 mg/L) on nitrification in activated sludge. 50 mg/L of ammonia was completely oxidized to nitrate in 10 h, but above 100 mg/L it remained in the

Table 1
Chemical compositions of the influent and effluent of nitrification process in a full-scale cokes wastewater treatment facility

	COD	Phenols	Total N	NH_4^+	NO_2^-	NO_3^-	SCN^-	Total CN
Influent (mg/L)	~400	~15	~200	~40	ND	ND	~80	~13
Effluent (mg/L)	300	ND	58	ND	2.59	38.0	ND	10.33
	Free CN^-	Total organic carbon	Inorganic carbon	PO_4^{3-}	SO_4^{2-}	F^-	Cl^-	Fe
Influent (mg/L)	<0.1	~120	~40	<5	~500	~100	~1000	~5
Effluent (mg/L)	ND	80.3	24.6	3.2	1035	89.4	1150	5.13

ND means 'Not Detected'.

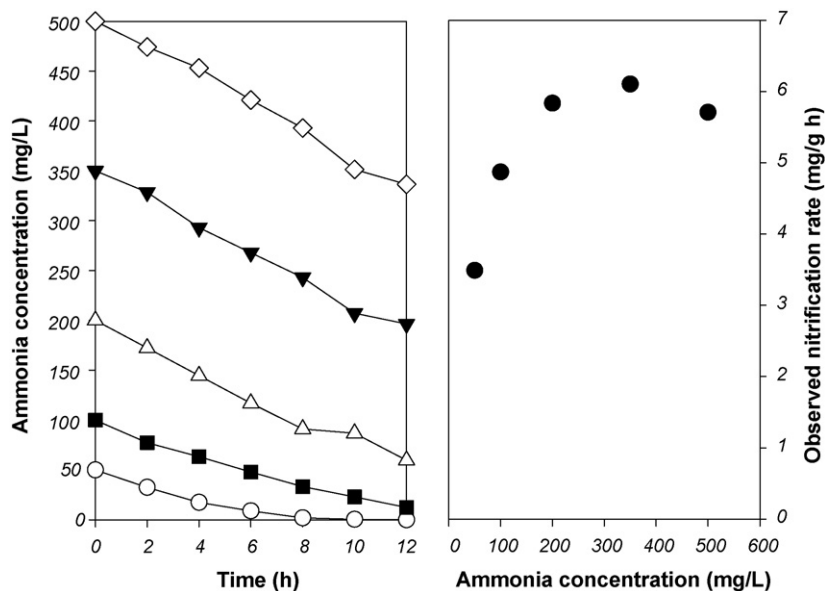


Fig. 2. Effect of initial ammonia concentration on nitrification in activated sludge.

solution at 12 h. To numerically examine the effect of ammonia concentration on nitrification, observed nitrification rates were calculated from declines of ammonia concentration. The nitrification rate increased with increasing ammonia concentration to 350 mg/L, and then declined apparently (Fig. 2). This result indicates that ammonia concentration below 350 mg/L did not inhibit microbial activity of nitrifying bacteria in our activated sludge system.

It is well known that inhibitory effect of ammonia on nitrification is due to not ammonia itself but free ammonia (FA), which can be formed at high concentration of total ammonia [13,17]. According to an equation proposed by Ford et al. [18], 350 mg/L of total ammonia contains 9.0 mg/L of FA at pH 7.5 and 30 °C.

$$\text{FA (mg/L)} = \frac{[\text{Total NH}_3] \times 10^{\text{pH}}}{\exp[(6334/(273 + T))] + 10^{\text{pH}}} \quad (1)$$

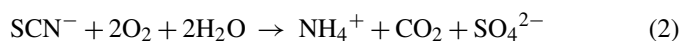
Previous researches reported that the threshold FA concentration for inhibiting *Nitrosomonas* and *Nitrobacter* were 10–150 mg/L and 0.1–4.0 mg/L, respectively [13,19–21]. In the case of 350 mg/L total ammonia concentration, therefore, the FA concentration was lower than threshold FA concentration for *Nitrosomonas*, but higher than that for *Nitrobacter*.

Although cokes wastewater usually contained above 100 mg/L of ammonia, about 40 mg/L of it flowed into nitrification process of a full-scale cokes wastewater treatment facility, as shown in Table 1. Therefore, there was no possibility of incomplete nitrification due to inhibitory effect of ammonia itself.

3.2. Effect of thiocyanate on nitrification

In addition to ammonia, thiocyanate is a major nitrogen compound to be removed, since cokes wastewater contains high concentration of it. To evaluate a threshold thiocyanate concentration for inhibiting nitrification, the effect of initial thiocyanate

concentration was examined in the range of 10–200 mg/L (Fig. 3a). 10 mg/L of thiocyanate concentration did not affect nitrification, but 200 mg/L of it slightly inhibited nitrification in activated sludge. During nitrification in activated sludge, 200 mg/L of thiocyanate was also completely removed in 18 h (data not shown). It is well known that activated sludge contains various autotrophic bacteria capable of degrading thiocyanate under aerobic condition according to the following equation [22].



48 mg-N/L of ammonia can be formed from 200 mg/L of thiocyanate theoretically. Thus, the decreased rate of nitrification might be due to the increased loading of ammonia, not toxicity of thiocyanate itself.

In full-scale pre-denitrification process of cokes wastewater treatment, most of thiocyanate flowed into aeration tank, since it could not be degraded under denitrification condition. Generally, influent thiocyanate concentration of nitrification process was below 100 mg/L due to dilution effect of recycled effluent (Table 1). Thus, there was no possibility of nitrification inhibition by toxic effect of thiocyanate itself. However, it should be pointed out that an abrupt increased loading of thiocyanate may cause incomplete degradation of it in activated sludge, resulting poor removal efficiency of total nitrogen. For this reason, influent thiocyanate concentration has been used as an indicator of total nitrogen compounds in cokes wastewater (Note that thiocyanate can be easily analyzed by a colorimetric method using ferric nitrate).

3.3. Effect of cyanide compounds on nitrification

Although toxic effect of free cyanide on biological nitrification has been fully researched [3,8,11,12,16], to the best of our knowledge, species of cyanide compounds present in cokes

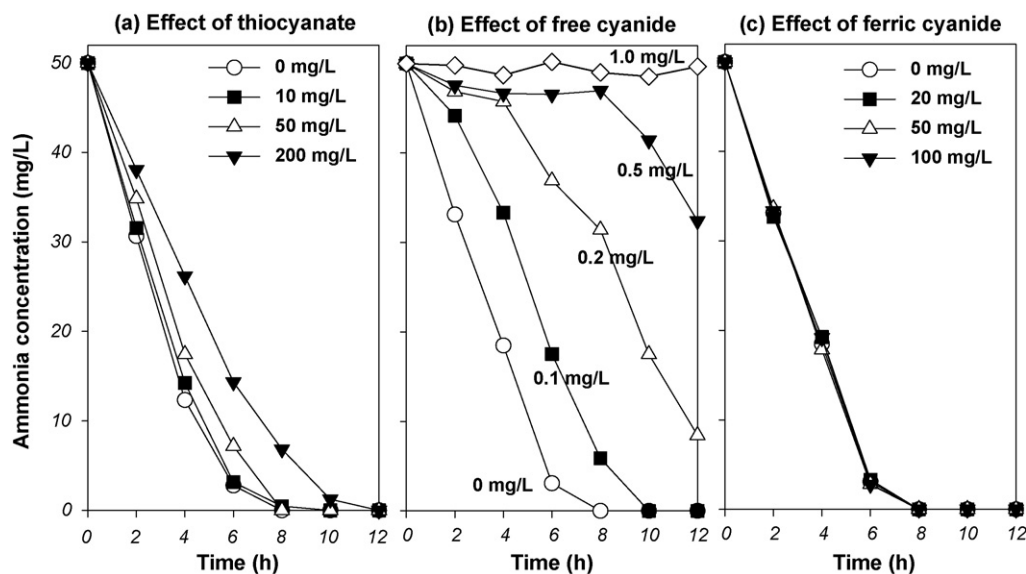


Fig. 3. Effects of (a) initial thiocyanate, (b) free cyanide, and (c) ferric cyanide concentrations on nitrification in activated sludge.

wastewater has been not considered [4,5,7–9]. Free cyanide can easily complex with various metals, and the metal-complexed cyanides are known to be less toxic than free cyanide or non toxic [23]. As shown in Table 1, cokes wastewater contained about 5 mg/L of iron in a form of ferric ion. Thus, cyanide compounds were present in the forms of free cyanide (CN^-) and ferric cyanide ($\text{Fe}(\text{CN})_6^{3-}$); the former ranged from 1 to 5 mg/L, the latter from 20 to 40 mg/L.

Fig. 3b shows the effect of initial free cyanide concentration on nitrification in activated sludge. 0.1 mg/L of free cyanide slightly reduced initial rate of nitrification, but 50 mg/L of ammonia was completely nitrified in 10 h. Free cyanide above 0.2 mg/L caused a distinct lag phase on nitrification in activated sludge. As free cyanide concentration increased, the length of lag phase increased and complete inhibition was reached at 1.0 mg/L of free cyanide. Similar results were reported by other researchers. Neufeld et al. [12] reported that maximum level of free cyanide to permit nitrification was 0.11 mg/L. Kim and Kim [16] reported that the increase of cyanide concentration (above 0.5 mg/L) produced excessive foam and decreased the microbial activity of activated sludge within the aeration tank, then the wastewater treatment process failed in operation due to poor settle-ability as well as sludge raising in a clarifier. Fortunately, free cyanide below 0.1 mg/L flowed into nitrification process of full-scale cokes wastewater treatment facility owing to its anaerobic degradation under denitrification condition (Table 1). According to a report of Richards and Shieh [24], free cyanide can be removed to some degree by anaerobic bacteria. Therefore, there was less possibility of incomplete nitrification by toxic effect of free cyanide under normal condition, but an abnormal influx of free cyanide into nitrification process may cause an irrecoverable damage to nitrifying bacteria.

Effect of ferric cyanide on nitrification was also examined in the range of 20–100 mg/L. As can be seen in Fig. 3c, increasing ferric cyanide concentration to 100 mg/L did not affect nitrification in activated sludge. This implies that ferric cyanide was

virtually a non-inhibitory compound to nitrifying bacteria. The difference in toxicity between free cyanide and ferric cyanide is owing to high stability of ferric cyanide [23].



Therefore, there is no need to consider inhibitory effect of ferric cyanide on the full-scale pre-denitrification process of cokes wastewater treatment, if considering its influent concentration (Table 1).

3.4. Effect of phenol compounds on nitrification

Among various organic compounds present in cokes wastewater, phenol compounds are known to negatively affect nitrifying bacteria [12,14,25,26]. Thus, the effects of various phenol compounds, such as phenol and *p*-cresol, on nitrification were examined in activated sludge system.

As shown in Fig. 4a, phenol negatively affected nitrifying activity in activated sludge. As initial phenol concentration increased, the nitrification rate decreased. Particularly, phenol above 200 mg/L significantly inhibited nitrification, and nitrification did not occur when phenol concentration was 500 mg/L. These results differed from results obtained from pure culture of nitrifying bacteria. Dyreborg and Arvin [25] reported that pseudo-critical concentration of phenol was 3.7 mg/L for nitrifying bacteria. Neufeld et al. [12] also observed inhibitory effects of phenol above 5.5 mg/L and coal tar acid mixtures (as phenol) above 1.7 mg/L. In comparison to these values, it is very interesting that the threshold phenol concentration for inhibiting nitrification was 100–200 mg/L in our activated sludge system. Fig. 4b shows dynamics of phenol concentration during nitrification in activated sludge. 200 mg/L of phenol was completely removed in 2 h owing to its rapid aerobic degradation. In the case of 500 mg/L phenol, however, half of it remained in the solution and caused serious inhibition on nitrification in acti-

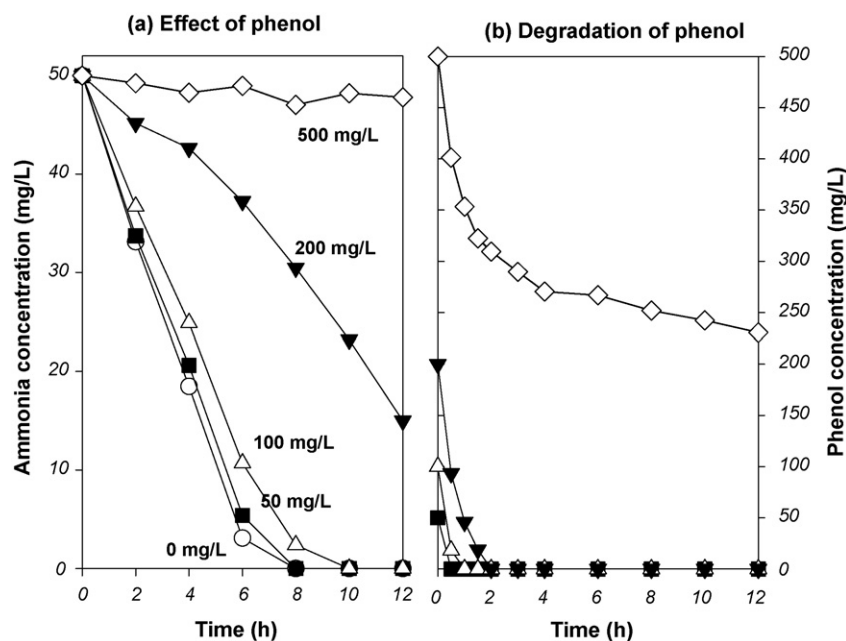


Fig. 4. (a) Effect of initial phenol concentration on nitrification and (b) its degradation in activated sludge.

vated sludge. Generally, it is well known that activated sludge in aeration tank contains various phenol-degrading bacteria [6,27]. After all, because of co-existence of phenol-degrading bacteria within the activated sludge used in this study, nitrification was not inhibited by phenol below 100 mg/L. As a matter of fact, phenol usually flowed into full-scale nitrification process below 20 mg/L owing to dilution effect and its partial degradation during denitrification (Table 1). Therefore, there was also no possibility of incomplete nitrification due to toxic effect of phenol itself. However, it has been reported that even low concentration of phenol may negatively affect full-scale nitrification process through an indirect mechanism, i.e., oxygen depletion due to fast growth of heterotrophic phenol-degrading bacteria [17].

Fig. 5 shows the effect of initial *p*-cresol concentration on nitrification in activated sludge. 20 mg/L of *p*-cresol did not affect nitrification rate, but it decreased with increasing initial *p*-cresol concentration. Texier and Gomez [28] reported that *p*-cresol at 17 mg/L inhibited by 70% nitrate formation in a nitrifying culture, whereas at 10 mg/L, there was no effect. Dyreborg and Arvin [25] estimated the pseudo-critical concentration of *o*-cresol at 1.3 mg/L. The reason for high threshold concentration (50 mg/L) of *p*-cresol in this study might be due to its biodegradation in activated sludge, like similar result by Kim and Kim [16]. They observed the inhibitory effect of *m*-cresol above 50 mg/L on nitrification in activated sludge.

3.5. Methods for reducing inhibitory effects of toxic compounds

Batch experiments with activated sludge showed that free cyanide and phenol may significantly inhibit the full-scale nitrification process of cokes wastewater treatment through direct and/or indirect mechanism(s). In order to operate this process

stably, therefore, it is very important to prevent an abrupt influx of these toxic compounds into the process or to reduce their inhibitory effects by using appropriate chemical and/or physical method(s).

It is known that activated carbon can remove various toxic compounds from aqueous solution [29]. Thus, the feasibility test of activated carbon for removing phenol and free cyanide was conducted in activated sludge system. When comparing the results in Fig. 4a, addition of activated carbon into activated sludge system could significantly reduce the inhibitory effect of phenol on nitrification (Fig. 6a). Nitrification was not at all inhibited by phenol below 200 mg/L and only slightly inhibited by 500 mg/L phenol. As seen in Fig. 6b, meanwhile, activated carbon addition also reduced inhibitory effect of free cyanide on nitrification in activated sludge, but less effective than phenol.

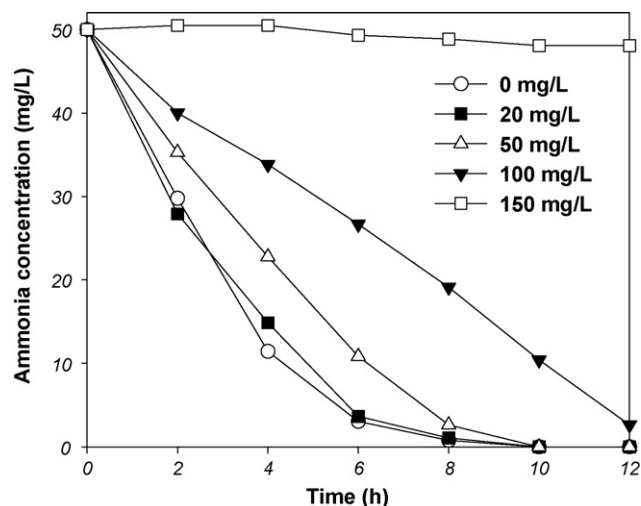


Fig. 5. Effect of initial *p*-cresol concentration on nitrification in activated sludge.

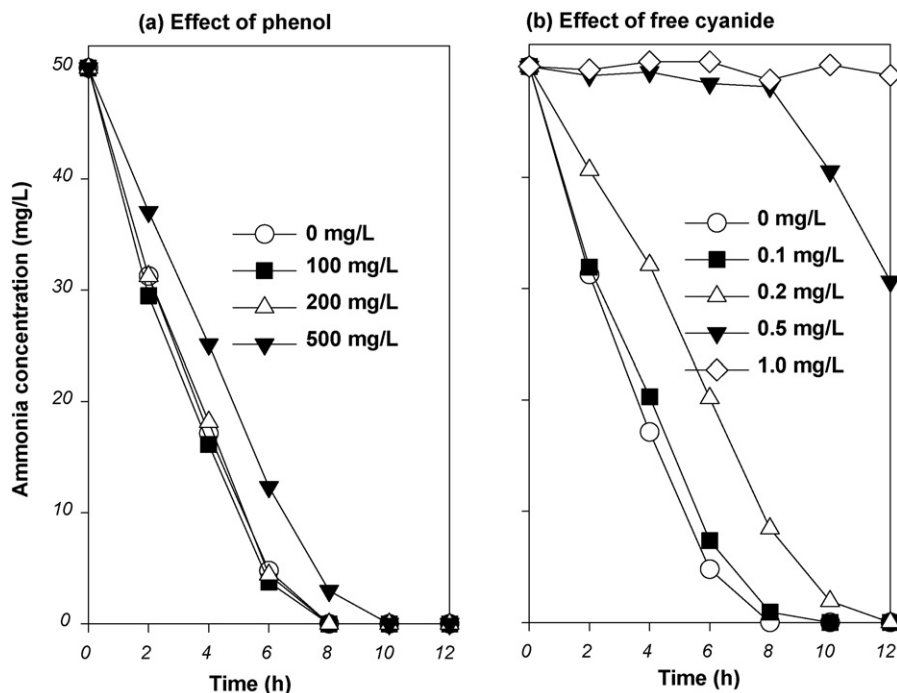


Fig. 6. Effects of (a) initial phenol and (b) free cyanide concentrations on nitrification in activated sludge added with 10 g/L of activated carbon.

This result was due to poor adsorption efficiency of activated carbon for free cyanide (data not shown). In short, activated carbon addition into aeration tank must be an effective method for reducing inhibitory effects of toxic compounds, especially phenol, on the full-scale pre-denitrification process for treating cokes wastewater. However, fresh activated carbon needs to be supplemented in the biological reactor and this may increase the treatment cost significantly. In order to decrease the cost activated carbon should be reused for several cycles by re-activation or bio-regeneration method.

4. Conclusions

In a full-scale pre-denitrification process for treating cokes wastewater, some major compounds present in the wastewater can cause inhibitory effect on microbial activity in activated sludge. Particularly, insufficient removal of total nitrogen can occur by inhibitory effects of toxic compounds on nitrification. Among various compounds, free cyanide most seriously inhibits nitrification in activated sludge, thus it should be flowed into aeration tank below its threshold concentration. Phenol compounds also negatively affect nitrification, but less than free cyanide. However, there is less possibility of nitrification inhibition by phenol itself owing to its complete biodegradation under anoxic and aerobic conditions. Besides, an abrupt increased loading of thiocyanate may cause its incomplete degradation in activated sludge, resulting poor removal efficiency of total nitrogen. In conclusion, the influx of toxic compounds into the full-scale pre-denitrification process should be controlled below their threshold concentrations through on-line or off-line monitoring of each compound. Inhibitory effects of toxic compounds

by an abnormal influx of those can be effectively reduced by activated carbon addition.

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