

## Effect of phenol on the biological treatment of wastewaters from a resin producing industry

M. Eiroa, A. Vilar, C. Kennes, M.C. Veiga

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### Abstract

The effect of phenol on the biological treatment of wastewaters from a resin producing industry was analyzed in a pre-denitrification system. First, the effect of phenol overloads on the removal of organic matter and nitrogen compounds was studied. During the overloads (from 250 to 4000 mg/L), phenol was detected in the effluent of the anoxic reactor but the system recovered fast after stopping the overloads. The total organic carbon (TOC) removal remained unchanged during phenol addition (91.9% at 0.20 kg TOC/m<sup>3</sup> d), except for the highest overload. With regard to total Kjeldahl nitrogen (TKN), its mean removal (87.9% at 0.08 kg TKN/m<sup>3</sup> d) was not affected by the phenol overloads. Afterwards, the effect of different phenol concentrations on the biological treatment of these wastewaters was analyzed. Phenol concentrations from 250 to 4000 mg/L were added to the feed. Phenol was completely removed despite the presence of other carbon sources in the wastewater. In spite of the presence of phenol, a TOC removal around 91.3% was achieved at an average organic loading rate of 0.11 kg TOC/m<sup>3</sup> d. The mean applied nitrogen loading rates were 0.05 and 0.08 kg TKN/m<sup>3</sup> d, obtaining TKN removals around 85.8% and 87.1%, respectively. Therefore, the biological treatment of wastewaters from a resin producing industry in a pre-denitrification system was not affected by the presence of phenol.

### Keywords

Phenol; industrial wastewaters; biological treatment.

# 1. Introduction

Wastewaters from resin producing industries are characterized by the presence of organic matter and nitrogen compounds. Consequently, the biological treatment of these wastewaters requires a combined process for carbon and nitrogen removal. This biological treatment could be carried out in a pre-denitrification system which avoids or decreases the need of an external carbon source, which is important from an environmental and economic point of view. Organic matter removal, hydrolysis of nitrogen compounds and denitrification of nitrate recirculated from the aerobic unit would take place in the anoxic reactor. Nitrification of ammonium provided by the anoxic unit and biodegradation of the organic matter that would not have been removed in the anoxic reactor would take place in the aerobic reactor.

Cheng et al. (1996) studied the treatment of wastewaters from a resin producing industry using a pre-denitrification system (anoxic–aerobic–aerobic) at lab scale. Efficiencies of chemical oxygen demand (COD) and total Kjeldahl nitrogen (TKN) removal around, respectively, 95.3% and 83.8%, were achieved at organic loading rates between 0.27 and 0.72 kg COD/m<sup>3</sup> d and nitrogen loading rates between 0.04 and 0.12 kg TKN/ m<sup>3</sup> d. Garrido et al. (2000) also analyzed the treatment of wastewaters from a resin producing industry using a pre-denitrification system (anoxic–aerobic) at lab scale. At organic loading rates from 0.7 to 1.9 kg COD/m<sup>3</sup> d, the COD removals were between 70% and 85%.

The composition of wastewaters from resin producing industries is dependent on the manufacturing process. Phenol is not typically present in these wastewaters but odd effluents with high concentrations of this compound are generated when the manufacturing process is changed. Therefore, it is necessary to study the effect of phenol on the biological treatment of these wastewaters. In spite of being a toxic compound there are several references about phenol biodegradation under both anoxic (Blaszczyk et al., 1998, Sarfaraz et al., 2004 and Eiroa et al., 2005) and aerobic conditions (Buitrón et al., 1998, González et al., 2001, Yamagishi et al., 2001 and Amor et al., 2005).

The aim of this work was to analyze the effect of phenol on the biological treatment of wastewaters from a resin producing industry in a pre-denitrification system. First, the effect of phenol overloads on the removal of organic matter and nitrogen compounds was studied. Afterwards, the effect of different phenol concentrations on the biological treatment of these wastewaters was analyzed.

## 2. Methods

### 2.1. Analytical methods

Phenol was determined using a Hewlett–Packard 1100 liquid chromatograph equipped with a C-18 ODS column (25 cm × 4 mm ID). The mobile phase was methanol:water (60:40) and detection was performed at 280 nm. Methanol was measured using a Hewlett–Packard 5890-II gas chromatograph equipped with a Nukol column (30 m × 0.25 mm ID) and a flame ionization detector. Nitrogen (1.5 mL/min) was utilised as carrier gas. Injector and detector temperatures were 250 and 270 °C,

respectively. Formaldehyde was analyzed spectrophotometrically according to the Hantzsch reaction (Nash, 1953), using a Perkin–Elmer Lambda 11 UV/Vis spectrophotometer. Total organic carbon (TOC) was determined according to *Standard Methods* (APHA et al., 1998) using a TOC-5050A Shimadzu.

Nitrite and nitrate were analyzed by capillary electrophoresis using a Hewlett–Packard <sup>3D</sup>CE system with a microcapillary tube of fused silica (40 cm × 50 μm ID). UV detection was undertaken at a wavelength of 214 nm and 450 nm as reference. The biogas composition (N<sub>2</sub>, CH<sub>4</sub>, CO<sub>2</sub> and N<sub>2</sub>O) was analyzed on a Hewlett–Packard 5890-II gas chromatograph equipped with a Porapak Q W80/100 column (2 m × 1/8” ID) and a thermal conductivity detector. Helium (15 mL/min) was utilised as carrier gas. Injector, oven and detector temperatures were 90, 25 and 100 °C, respectively. Ammonium, pH, total Kjeldahl nitrogen (TKN) and volatile suspended solids (VSS) were evaluated according to *Standard Methods* (APHA et al., 1998).

## 2.2. Lab-scale reactor

The pre-denitrification system (Fig. 1) consisted of an anoxic upflow sludge blanket reactor (0.8 L) and an aerobic activated sludge reactor (1.8 L) (Eiroa et al., 2006). The system was provided with a liquid displacement biogas measurement device (Veiga et al., 1990). The feed to the anoxic reactor was supplied by a peristaltic pump and its effluent was continuously fed to the aerobic reactor. Diffusers, located at the bottom of the aerobic reactor, supplied air from an air pump and maintained complete mixing. The water was separated from the sludge in the settler and the sludge was recycled intermittently to the aeration basin. Part of the effluent of the aerobic unit was recirculated to the anoxic reactor by another peristaltic pump.

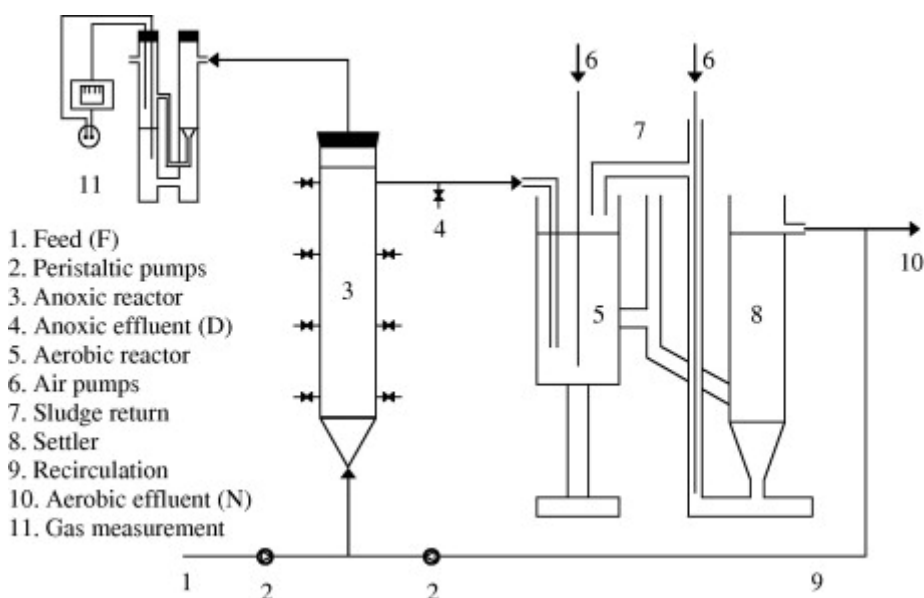


Fig. 1.

Scheme of the pre-denitrification system.

The reactors were inoculated with sludge from the full-scale wastewater treatment plant of a resin producing industry. Assays were performed in a thermostatic chamber at

20 °C. The feed consisted of wastewaters obtained at different times from the industry mentioned above; the composition of these wastewaters is shown in Table 1.

Table 1.

Composition of wastewaters from a resin producing industry (all parameters are in mg/L, except pH)

Parameter	Wastewater I		Wastewater II-a		Wastewater II-b	
	Range	Average	Range	Average	Range	Average
CH <sub>3</sub> OH	2642.7– 2688.1	2660.0	1335.6– 1347.2	1340.0	1332.9– 1343.4	1340.0
CH <sub>2</sub> O	4.8–22.3	14.7	3.5–7.1	4.9	22.6–29.1	26.4
C <sub>6</sub> H <sub>6</sub> O	3.3–4.4	3.9	–	–	–	–
TOC	1243.5– 1513.0	1360.0	695.0– 716.1	704.1	759.4– 782.4	771.8
TKN	532.0– 559.7	544.7	306.4– 362.6	327.3	495.3– 550.7	519.9
N – NH <sub>4</sub> <sup>+</sup> N- NH <sub>4</sub> <sup>+</sup>	328.9– 372.7	348.5	193.7– 229.5	205.4	255.7– 300.0	282.7
N – NO <sub>3</sub> <sup>-</sup> N- NO <sub>3</sub> <sup>-</sup>	0.1–1.2	0.6	0.0–0.9	0.2	7.0–9.0	7.7
pH	8.2–9.1	8.6	7.3–8.4	8.0	7.0–8.6	7.8

The concentrations of the different parameters in the feed (F) and in the effluent of the anoxic (D) and aerobic reactors (N) are presented in the figures. Mass balances with regard to the feed were performed in order to calculate the removal percentages of the different compounds in the global system and in each individual unit.

## 3. Results and discussion

### 3.1. Effect of phenol overloads

The effect of phenol overloads on the biological treatment of wastewater from a resin producing industry was analyzed in order to simulate the effect of odd effluents with high concentrations of phenol. For this study, the system was fed wastewater I which had the composition shown in Table 1. The feed was supplied at a rate of 0.38 L/d and 3.7 L/d of the aerobic effluent were recirculated to the anoxic reactor. The total hydraulic retention time was 6.8 days, corresponding to 2.1 days in the anoxic reactor and 4.7 days in the aerobic reactor. The anoxic and aerobic reactors were inoculated with 7.5 and 4.0 g VSS/L, respectively.

#### 3.1.1. Biological removal of organic matter

The inoculum of the pre-denitrification system had been acclimatized to the industrial wastewater through several months. The system was fed wastewater I during the first 17

days of operation, and afterwards phenol overloads were applied. During the first 17 days of operation, the TOC concentration in the feed was between 1243.5 and 1513.0 mg/L, corresponding to an average organic loading rate of 0.20 kg TOC/m<sup>3</sup> d. The TOC values in the effluent of the aerobic reactor became stable around 105.0 mg/L (Fig. 2b), being the mean removal of 91.9%. This is in agreement with the results obtained by our group in a similar study with wastewater from a resin producing industry which contained high levels of formaldehyde and formic acid (Eiroa et al., 2006), where the TOC values in the feed were between 1423.0 and 1599.5 mg/L, corresponding to an organic loading rate of about 0.20 kg TOC/m<sup>3</sup> d. A high TOC removal was also achieved, around 92.0%.

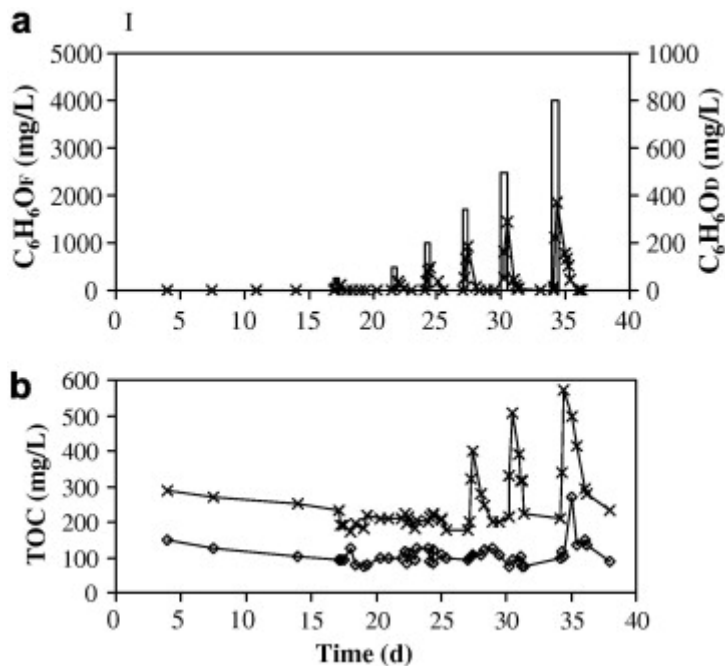


Fig. 2.

(a) Phenol concentration in the feed (—) and in the effluent of the anoxic reactor (×). (b) TOC in the effluent of the anoxic (×) and aerobic (◇) reactors (wastewater I).

Figure options

The methanol concentration in the feed was around 2660.0 mg/L, corresponding to an organic loading rate of 0.15 kg C/m<sup>3</sup> d. The average methanol removal was 99.8%; its removal took mainly place in the anoxic reactor. This high methanol removal was expected due to its wide use as external carbon source (Nyberg et al., 1992) since it is readily assimilated for denitrifying bacteria. In addition to methanol, other organic compounds were analyzed. The formaldehyde concentration in the feed ranged between 4.8 and 22.3 mg/L, being its mean value in the aerobic effluent of 0.9 mg/L. With regard to phenol, its concentration in the feed was also very low (between 3.3 and 4.4 mg/L) and it was totally removed.

### 3.1.2. Biological removal of nitrogen compounds

The removal of nitrogen compounds was also studied in wastewater I. The TKN concentrations in the feed and in the effluent of the anoxic and aerobic reactors are shown in Fig. 3. The TKN concentration in the feed ranged between 532.0 and 559.7 mg/L, obtaining a mean value in the aerobic effluent of 62.8 mg/L. The percentage of TKN removal was 87.9%, at a nitrogen loading rate around 0.08 kg TKN/m<sup>3</sup> d.

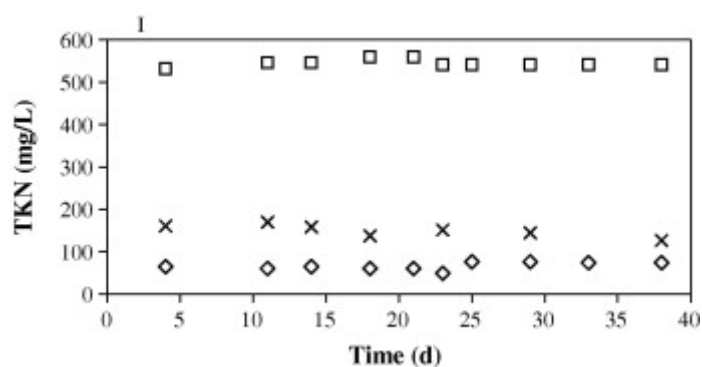


Fig. 3.

TKN (□) in the feed and in the effluent of the anoxic (×) and aerobic (◇) reactors (wastewater I).

The nitrate concentration in the feed was very low, with a mean value of 0.6 mg N/L. In the aerobic unit the ammonium from the anoxic reactor was nitrified and assimilated by microorganisms, being the ammonium removal around 99.5%. Meanwhile, the denitrification of the nitrate recirculated from the aerobic unit took place in the anoxic reactor, obtaining average removal efficiencies of 99.8%. The biogas produced in the anoxic reactor was periodically analyzed and was basically composed of nitrogen (around 95.4%), being the percentages of carbon dioxide and methane very low (around 1.1% and 0.1%, respectively).

In a similar study with wastewater from a resin producing industry which contained high levels of formaldehyde and formic acid (Eiroa et al., 2006), the TKN concentration in the feed was around 477.8 mg/L. The average applied nitrogen loading rate was 0.06 kg TKN/m<sup>3</sup> d, being the mean percentage of TKN removal of 76.7%. Since the ammonium concentration was very low (14.9 mg N/L), the TKN removal basically depended on the efficiency of the hydrolysis of organic nitrogen. Therefore, the TKN removal was lower than in this case (87.9% at 0.08 kg TKN/m<sup>3</sup> d). This can be due to the fact that most of the TKN was present as ammonium, around 348.5 mg N/L (Table 1). Consequently, in this case the TKN removal depended to a lesser extent on the hydrolysis of organic nitrogen.

Cheng et al. (1996) also studied the treatment of wastewaters from a resin producing industry using a pre-denitrification system (anoxic–aerobic–aerobic). The average TKN concentration in the feed was 389 mg/L (0.09 kg TKN/m<sup>3</sup> d) and most of the TKN was also present as ammonium, around 232 mg N/L. In spite of the different composition of the wastewaters, they achieved a percentage of TKN removal similar to the present study, around 83.8%.

### 3.1.3. Phenol overloads

After day 17 of operation, the system was still fed wastewater I but applying phenol overloads of 250, 500, 1000, 1700, 2500 and 4000 mg/L during 10 h each. Therefore, the applied phenol loading rate varied between 0.04 and 0.59 kg C<sub>6</sub>H<sub>6</sub>O/m<sup>3</sup> d (0.03–0.45 kg C/m<sup>3</sup> d). The phenol concentration in the feed and in the effluent of the anoxic reactor is shown in Fig. 2a. Phenol concentration in the effluent of the aerobic reactor is not shown because this compound was not detected at any of the different overloads, except for the highest one.

During the overloads, phenol was detected in the effluent of the anoxic reactor but the system recovered rapidly after stopping the overloads. In the effluent of this reactor a concentration of 21.0 mg/L phenol was reached for the lowest overload (250 mg/L) and 370.2 mg/L for the highest overload (4000 mg/L), although it disappeared in less than two days in all cases. In the effluent of the aerobic reactor phenol was only detected for the overload of 4000 mg/L, being the highest undegraded concentration 128.1 mg/L, although it disappeared in one day. Therefore, the pre-denitrification system returned to the pre-overload state in less than two days at all the applied overloads. In the case of an odd effluent with a high concentration of phenol, the anoxic reactor would not get destabilized, but part of the phenol would remain undegraded in this reactor, and would go to the aerobic unit. Finally, this residual phenol would be removed in the aerobic reactor, causing the proliferation of heterotrophic bacteria which would compete with nitrifying bacteria.

The TOC removal remained unchanged during the phenol overloads with regard to the first 17 days of operation, except for the highest overload (Fig. 2b). This is due to the fact that phenol was only detected in the effluent of the aerobic reactor during the highest overload of 4000 mg/L, reaching TOC concentrations of up to 270.1 mg/L. During the rest of the operation period, the TOC values in the effluent of the aerobic reactor ranged between 72.4 and 127.1 mg/L. With regard to nitrogen compounds, the mean TKN removal was not affected by the phenol overloads, obtaining the same TKN concentrations in the aerobic effluent than during the first 17 days of operation (Fig. 3). The ammonium removal and denitrification percentages remained also unchanged during the phenol overloads.

## 3.2. Effect of different phenol concentrations

Since the removal of organic matter and nitrogen compounds was not affected by the phenol overloads, the effect of different phenol concentrations on the biological treatment was analyzed. For this study, the system was fed wastewaters II-a and II-b obtained from a resin producing industry (Table 1). The feed was supplied by a peristaltic pump at a flow rate of 0.38 L/d, with a recirculation from the aerobic to the anoxic unit at a rate of 3.7 L/d. The total hydraulic retention time was 6.8 days, corresponding to 2.1 days in the anoxic reactor and 4.7 days in the aerobic unit. The anoxic and aerobic reactors were inoculated with 7.5 and 3.0 g VSS/L, respectively.

### 3.2.1. Biological removal of organic matter

The organic compound present in the wastewaters at high concentrations was methanol. Its concentration in the feed was around 1340.0 mg/L, corresponding to an organic

loading rate of  $0.07 \text{ kg C/m}^3 \text{ d}$ , and it was completely removed during all the operation period. As in wastewater I, methanol removal took mainly place in the anoxic reactor. Formaldehyde was also detected in the wastewaters but at very low concentrations (between 3.5 and 29.1 mg/L), being its mean value in the aerobic effluent of 0.3 mg/L. Formaldehyde was almost completely removed in the anoxic reactor as well. In other studies formaldehyde was also completely removed in an anoxic reactor in the presence of phenol (Eiroa et al., 2005).

Different phenol concentrations were added to the feed (250, 500, 1000, 1700, 2500 and 4000 mg/L) in order to study the effect on the biological treatment of this wastewaters (Fig. 4). Therefore, the applied phenol loading rate varied between 0.04 and  $0.59 \text{ kg C}_6\text{H}_5\text{O/m}^3 \text{ d}$  ( $0.03\text{--}0.45 \text{ kg C/m}^3 \text{ d}$ ). In spite of the presence of other carbon sources in the wastewater, phenol was completely removed at all concentrations. The phenol concentration in the effluent of the aerobic reactor remained always under the detection limit (lower than 0.1 mg/L). Phenol was almost completely removed in the anoxic reactor, but part of it went to the aerobic unit. Finally, this part of the phenol was removed in the aerobic reactor, causing the proliferation of heterotrophic bacteria. The presence of organic matter stimulates the growth of heterotrophic bacteria, which compete with nitrifying bacteria for their basic substrates, ammonium and oxygen (Hanaki et al., 1990). Consequently, competition between nitrifying and heterotrophic bacteria could exist in the aerobic unit.

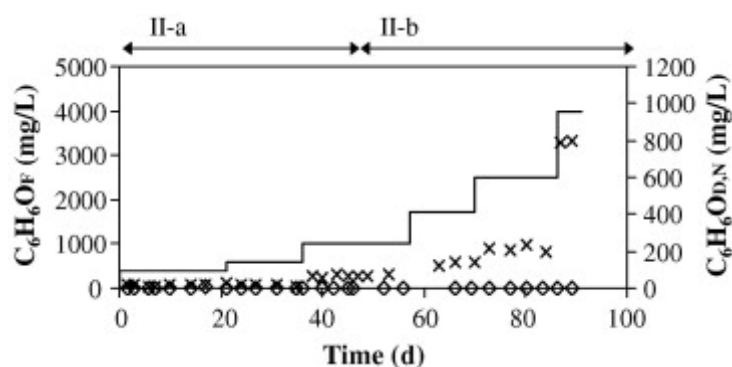


Fig. 4.

Phenol concentration in the feed (—) and in the effluent of the anoxic (×) and aerobic (◇) reactors (wastewater II).

Data of phenol biodegradation at higher organic loading rates have been reported using synthetic wastewater under anoxic conditions. Blaszczyk et al. (1998) studied phenol biodegradation under denitrifying conditions in a packed reactor. The highest biodegradation rate ( $2.5 \text{ kg C}_6\text{H}_5\text{O/m}^3 \text{ d}$ ) was obtained using a phenol concentration in the influent of 200 mg/L and operating the reactor at a hydraulic retention time of 2 h. Sarfaraz et al. (2004) analyzed phenol removal under anoxic conditions using a sequencing batch reactor. They achieved more than 80% phenol removal for concentrations up to 1050 mg/L ( $2.7 \text{ kg C}_6\text{H}_5\text{O/m}^3 \text{ d}$ ). However, when the concentration was increased to 1150 mg/L, phenol removal efficiency decreased to 56% ( $3.1 \text{ kg C}_6\text{H}_5\text{O/m}^3 \text{ d}$ ). Eiroa et al. (2005) studied phenol biodegradation in the presence of formaldehyde in an anoxic upflow sludge blanket reactor. Phenol removal efficiencies above 90.6% were obtained at phenol concentrations in the influent up to 755 mg/L ( $0.42 \text{ kg C}_6\text{H}_5\text{O/m}^3 \text{ d}$ ). However, when the phenol concentration was increased to 1010 mg/L ( $0.56 \text{ kg C}_6\text{H}_5\text{O/m}^3 \text{ d}$ ), its removal efficiency decreased.



At an average organic loading rate of 0.11 kg TOC/m<sup>3</sup> d, a TOC removal around 91.3% was obtained. Thus, in spite of the addition of high phenol concentrations, the efficiency of TOC removal was very high. The TOC values in the effluent of the aerobic reactor were between 65.5 and 171.4 mg/L, being the mean value 118.2 mg/L. Therefore, the removal of organic matter was not affected by the presence of phenol.

### 3.2.2. Biological removal of nitrogen compounds

The effect of different phenol concentrations on the removal of nitrogen compounds was also studied. The average TKN concentration in the feed was 327.3 mg/L in wastewater II-a and 519.9 mg/L in wastewater II-b (Fig. 5). Most of the TKN was present as ammonium, at a concentration around 205.4 mg N/L in wastewater II-a and 282.7 mg N/L in wastewater II-b. The mean applied nitrogen loading rate was 0.05 and 0.08 kg TKN/m<sup>3</sup> d, respectively. The TKN values reached in the aerobic effluent with wastewater II-a were between 10.8 and 82.4 mg/L, being the average TKN removal 85.8%. When the feed was changed to the wastewater II-b, the TKN in the effluent of the aerobic reactor increased up to 190.3 mg/L. After several days of operation with wastewater II-b, the TKN values obtained in the aerobic effluent were around 64.3 mg/L, being the average TKN removal 87.1%. The removal percentage was similar in both cases; however the TKN in the aerobic effluent was high with wastewater II-b because of the high organic nitrogen concentration in this wastewater. The hydrolysis of organic nitrogen was not complete in the pre-denitrification system. Different post treatments are being evaluated with wastewater from the same resin producing industry in order to decrease the residual TKN.

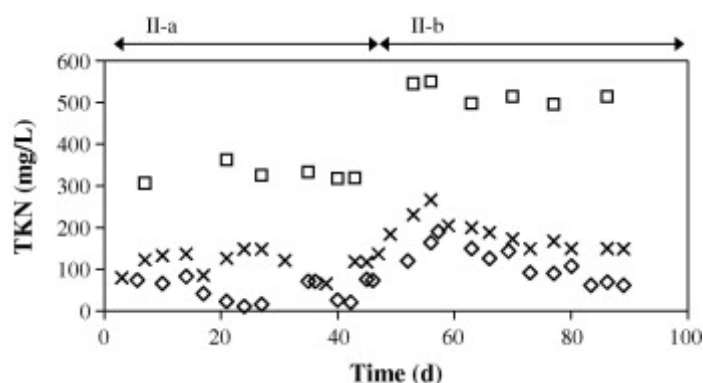


Fig. 5.

TKN (□) in the feed and in the effluent of the anoxic (×) and aerobic (◇) reactors (wastewater II).

The nitrate concentration in the feed was very low, between 0.0 and 9.0 mg N/L. An average ammonium removal of 99.8% was obtained and the efficiency of the denitrification process was above 99.9%. Therefore, the removal of nitrogen compounds was not affected by the presence of high concentrations of phenol (up to 4000 mg/L). The composition of the biogas produced in the anoxic system was periodically analyzed, obtaining percentages of nitrogen, carbon dioxide and methane between 73.9–98.2%, 0.0–2.2% and 0.0–16.6%, respectively. Consequently, denitrification and methanogenesis occurred simultaneously in the same unit. Previous studies indicate that if there is enough carbon source in the influent, both processes can occur in the same system (Chen and Lin, 1993).

## 4. Conclusions

Phenol overloads from 250 to 4000 mg/L (0.04–0.59 kg C<sub>6</sub>H<sub>6</sub>O/m<sup>3</sup> d) were applied to the pre-denitrification system treating wastewater from a resin producing industry. During the overloads, phenol was detected in the effluent of the anoxic reactor but the system recovered in less than two days after stopping the overloads. Therefore, in the case of an odd effluent with a high concentration of phenol, the pre-denitrification system would not get destabilized.

At an average organic loading rate of 0.20 kg TOC/m<sup>3</sup> d, a TOC removal around 91.9% was obtained. The mean TKN removal was 87.9%, at a nitrogen loading rate around 0.08 kg TKN/m<sup>3</sup> d. The TOC removal remained unchanged during phenol addition, except for the highest overload. With regard to TKN, its mean removal was not affected by the phenol overloads.

Phenol concentrations from 250 to 4000 mg/L (0.04–0.59 kg C<sub>6</sub>H<sub>6</sub>O/m<sup>3</sup> d) were added to the wastewaters from a resin producing industry. In spite of the presence of other carbon sources in the wastewater, phenol was completely removed at all concentrations. Phenol removal took mainly place in the anoxic reactor. At an average organic loading rate of 0.11 kg TOC/m<sup>3</sup> d, a TOC removal around 91.3% was obtained. Thus, in spite of the addition of high phenol concentrations, the efficiency of TOC removal was very high. The mean applied nitrogen loading rates were 0.05 and 0.08 kg TKN/m<sup>3</sup> d, obtaining TKN removals around 85.8% and 87.1%, respectively. The removal of nitrogen compounds was not affected by the presence of phenol.

Therefore, the biological treatment of wastewaters from a resin producing industry was not affected by the presence of phenol. Consequently, odd effluents with high concentrations of phenol can be biologically treated in a pre-denitrification system.

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