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Araştırma Makalesi Research Article

TREATABILITY OF OPIUM ALKALOID INDUSTRY WASTEWATERS BY ANAEROBIC PROCESSES

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Keywords

Opium alkaloid containing wastewater,
Anaerobic treatment,
Inhibition,

Abstract

The effluents from alkaloid industry contain high levels of organic matter, alkalinity, salinity, color and sulphate. The treatment of this complex industrial wastewater was attempted in two stages anaerobic reactors. The effects of COD, conductivity, pH, alkalinity, sulphate and TKN on anaerobic treatment were evaluated. When the reactors were fed with original wastewater, VFA's were accumulated in the presence of 25,794 mg/L COD and 30.2 mS/cm conductivity of the raw opium wastewater even at OLR of 1-3 kgCOD/m^{3}day. However, stepwise dilution was applied and COD removal of 90% was achieved at OLR of 7-8kgCOD/m^{3}day as the conductivity and COD were reduced to 20 mS/cm 20,000 mg/L, respectively. Based on the findings of this study, highly concentrated wastewaters of opium alkaloid industry wastewaters can be treated with anaerobic digestion and significant reduction of COD can be achieved.

AFYON ALKOLOİDLERİ ENDÜSTRİSİ ATIKSULARININ ANAEROBİK PROSESLERLE ARITILABİLİRLİĞİ

Anahtar Kelimeler

Afyon alkaloid çıkış suyu
Anaerobik arıtma,
İnhibisyon,

Öz

Alkaloid Endüstrisi çıkış suları yüksek konsantrasyonlu organic madde, alkalinite, tuzluluk, renk, sülfat içerir. Bu çalışmada sözkonusu kompleks endüstriyel atıksularının arıtımı, iki kademeli anaerobik rektörlerlerde denenmiştir. KOİ, iletkenlik, pH, alkalinite, sülfat, TKN'nin anaerobik arıtmaya etkileri değerlendirilmiştir. Reaktörler, 25,794 mg/L KOİ ve 30.2 mS/cm iletkenliğe sahip ham alkaloid endüstrisi çıkış suyu ile doldurulduğunda, OLR 1-3kgCOD/m^{3}gün iken bile uçucu yağ asitlerinin birikimi tespit edilmiştir. Ancak seyreltme yapılmış ve iletkenlik 20 mS/cm'e ve KOİ 2000 mg/L'e düşürülmüştür bu durumda OLR 7-8kgCOD/m^{3}gün'e çıkarılmış ve %90 oranında COD giderimi başarılmıştır. Bu çalışma ile, yüksek konsantrasyon içeren alkaloid endüstrisi atıksularının anaerobik çürütme ile arıtılabilirliği ve önemli ölçüde KOİ arıtımı sağlanabildiği görülmüştür.

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1. Introduction

Industrial processes generate polluted wastewaters which creat major environmental concerns and require treatment before discharging into receiving waters. In order to satisfy the environmental regulations, industrial organizations should consider establishing successful wastewater treatment processes. As an important example, the high strength effluents from opium alkaloid processing plants have been generating problem for years due to complex structural compounds contained in the wastewater.

Opium, a medicinally important compound, contains more than 40 individual alkaloids (opiates) such as morphine, codeine, thebaine, narcotine, and papaverine (Dereli et al., 2010). The cultivation of poppies and the processing of medicinal opium are practiced in a limited number of countries such as India, Australia, France, Hungary, Spain, and Turkey under the UN control (Baser and Arslan, 2014). Therefore, a limited number of researchers have worked on the treatment of alkaloid wastewaters so far.

Physicochemical treatment studies on the effluents of opium wastewater treatment plant were performed by Kınlı, (2004). It was reported that 70% chemical oxygen demand (COD) removal with anaerobic treatment was achieved. In addition, ozone oxidation was used during the pre-treatment of the aerobically treated effluents with color and COD removal efficiencies of 87% and 30%, respectively. In addition, 70% COD removal was reached by pilot scale upflow anaerobic sludge blanket reactor experiments (Sevimli et al., 1999). The anaerobic treatability of the mixture of the wastewaters generated from the distillation column and the domestic wastewater of an alkaloid plant was also performed by Ozturk et al. (2008). They determined COD and volatile fatty acid (VFA) removal efficiencies of 85 and 95%, respectively.

Aytimur and Atalay (2004) used activated sludge system for biological treatment with 88% COD removal and the catalytic wet air oxidation process for the chemical oxidation with the COD removal efficiency of 35%. They investigated the effectiveness of the individual processes and their combination. They concluded that the combinations of these processes are not very effective, however the biological treatment alone was sufficient.

Koyuncu (2003) tried membrane treatment as a post treatment and studied the COD and conductivity removals. It was reported that they were higher than 95%. Bural et al. (2010) studied the effect of irradiation during aerobic biological wastewater treatment. A set up with two sequencing batch reactors (SBR) were used in the experiments, one of the reactors was fed with raw wastewater and the other with wastewater irradiated by 40 kGy. The reactors were fed gradually increasing COD concentrations to acclimatize the biomass to the opium alkaloid wastewater. At 5000 mg/L COD concentration, the COD removal efficiencies of 79% and 73% were obtained for the reactors fed with raw and irradiated wastewaters, respectively.

Aydın et al. (2010) investigated the treatability of the opium industrial wastewaters by anaerobic processes. The COD concentration of the wastewater was varied between 5500 to 11700 mg/L and the COD removal efficiencies of 87.2% and 75% were achieved, respectively. They also identified some toxic organic chemicals such as N,N-dimethylaniline and toluene in the opium wastewaters. These compounds are known to be inhibitory for biological treatment processes. Cengiz et al (2017) worked on the hydrothermal gasification of opium alkaloid wastewater and found 95% COD removal at 600 ºC in the presence of a catalyst at the levels of 0.375–0.625 g.

Even though, the SO_4^{-2} content of the alkaloid industry wastewaters has not been determined, Ozdemir, 2006 reported sulphuric acid usage of 48.3 kg per ton of opium processed. Sakar et al. (2016) investigated the removal of sulfate from the nanofitration (NF) concentrate of alkaloid wastewater by electrodialysis. They concluded that the 99% of the sulfate retained in membrane concentrate in NF90 and NF245 membranes.

In the previous studies, mainly COD and biological oxygen demand (BOD) removals were studied in the experiments by using the diluted alkaloid industry wastewaters. However, these effluents are known to contain high concentrations of organic matter, alkalinity, salinity and SO_4^{-2}. In this project, the effects of COD, conductivity, pH, alkalinity, SO_4^{-2}, total Kjeldahl nitrogen (TKN) on anaerobic treatment of diluted and raw wastewaters were studied.

Anaerobic treatment of industrial wastewaters is generally carried out at dilute concentrations due to sensitivity of methanogenic consortia. This requires dilution of inlet wastewater. However, the trend is to carry out wastewaters as concentrated as possible due to lower volume requirement of anaerobic reactors and less water for dilution. The opium alkaloid industry wastewaters are highly complex and contain especially high concentrations of sulfate, and salinity that are toxic to biological treatment. The aim of this work to evaluate the treatability of opium industrial wastewater by anaerobic process at high inlet COD concentrations in view of not using dilution water or very little due to water scarcity which was not reported on an opium industrial wastewater treatment study by any researcher.

2. Materials and Methods

2.1. The Wastewater samples

The wastewater samples were obtained from an opium alkaloids factory in Turkey. The samples were collected in 30L containers and refrigerated at 4°C. The experiments were carried out over a nine-month period following the collection.

2.2. The Laboratory Scale Set-up

The study was carried out using a laboratory scale anaerobic digester (W8) consisting of two separate reactors (Armfield Ltd, UK). The main reason for having two stages was separating acid formation from methane production and determination of optimum conditions for each stage. The experimental set-up consists of two separate high-rate digesters with the total volume of 5 L and working volume of 4.8 L. The flow rates into the vessels were set and controlled by calibrated peristaltic pumps. The temperature of each reactor was kept at 37°C by an electric heating mat wrapped around the external wall of the reactors.

2.3. Anaerobic Treatment Experiments

The anaerobic sludges used in the upflow anaerobic reactors obtained from "Pakmaya Izmit Anaerobic Treatment Plant" and "ISKI Tuzla Municipal Wastewater Treatment Plant Sludge Digester". Both sludges were mixed equally and a 2.8 L of the mix was fed to each reactor. To create biofilm in each reactor, 100 balls shaped filling materials with 25 mm diameter and "LEVAPOR" biocarrier cubes were used. The C:N:P ratio of the feed was adjusted to 300:5:1 by the addition of phosphorus.

In each experimental run, the reactor was continuously operated under the different feed concentrations and loading rates until steady-state conditions were reached. Then samples were collected and analyzed.

2.3.1. Analysis

Total and soluble chemical oxygen demand (COD), volatile fatty acids (VFA), biochemical oxygen demand (BOD) total Kjeldahl nitrogen (TKN), total phosphorus (TP), total suspended solids (TSS), total volatile solids (TVS), color, alkalinity, sodium (Na), magnesium (Mg), calcium (Ca), sulphate (SO_4^{-2}) and ammonium (NH_4^+). pH, and conductivity in the the raw and treated opium industry wastewater were analyzed according to the American Public Health Association (2012) (Table 1).

Table 1. Analytical Methods Used to Measure Raw and Treated Opium Wastewater

Constituent	Analytical Method
COD	Standard Method 5220, a colorimetric, reactor digestion
BOD	Standard Method 5210 B
Conductivity and pH	YSI 6600 V2 model CTD
VFA	Standard Method 2540 G
Cations	ICP-MS, EPA 6020 A
NH4+	Standard Method 4500-NH3 H
SO4-2	Standard Method 4110 B, Dionex Ion Chromatography
TKN	Standard Method 4500 Norg B
Color	Standard Method 2120 B
SS	Standard Method 2540 D
VSS	Standard Method 2540 G
TDS	Standard Method 2540 C

3. Results and Discussion

3.1. The characterization of the opium industry wastewater

The characteristics of the opium alkaloid industry effluents used in the experiments and the cation profiles of the raw wastewater are illustrated in Table 2 and Table 3, respectively. The concentrations vary due to the combination of the production processes daily. Therefore, the average concentrations and standard deviations of various samples are given in Table 2.

Table 2. The characterization of opium processing industry effluents

Parameters	Unit	Concentration
pH		4.7±0.6
Biochemical oxygen demand (BOD_5)	(mg/L)	10151±3908
Soluble chemical oxygen demand (COD)	(mg/L)	32995.±8811
Suspended Solid (SS)	(mg/L)	427.5±235
Volatile Suspended Solids (VSS)	(mg/L)	426±217.5
Total Dissolved Solids (TDS)	(mg/L)	41133±6559
Inorganic TDS	(mg/L)	15910±8584
Conductivity	(µs/cm)	29750±10111
Volatile Fatty Acids (VFA)	(mg/L)	5806±1773
Total Kjeldahl Nitrogen (TKN)	(mg/L)	561±71
Ammonium Nitrogen (NH_4-N)	(mg/L)	22±5.6
Sulphate (SO_4^{-2})	(mg/L)	11656±4087
Total Phosphorus (TP)	(mg/L)	23.8±6.7
Alkalinity	(mg/L CaCO3)	4504±1866
Color	(PtCo)	2500

Table 3. The Cation Profile of the Raw Wastewater by ICP MS (Inductively Coupled Plasma – Mass Spectrometer)

Cations	Average Concentration (ppb)
Sodium	7192000
Potassium	5463000
Calcium	39010
Magnesium	19500
Iron	5131
Rubidium	3363
Nickel, Vanadium, Aluminum	500-1000
Copper, Zinc, Chromium, Stronsium, Scandium	100-500
Cobalt, Manganese, Lithium, Barium, Selenium, Cesium, Arsenic, Lead, Uranium,	1-100
Yttrium, Gallium, Lanthanum,	
Cerium, Praseodymium, Neodymium, Samarium,	
Europuium, Gadolinium,	
Terbium, Dysprosium,	
Holmium, Erbium, Thulium, Ytterbium, Lutetium, Rhenium, Thallium, Bismuth,	
Thorium, Indium, Beryllium, Cadmium	<5
Cations	Average Concentration (ppb)
Sodium	7192000

The high concentration of sodium is determined to originate from the process but the other cations represented in Table 3 are from the poppy straw. In addition, the source of the high concentration of potassium is determined to be the fertilizers used in ~~the~~ poppy fields (Copur et al., 2005).

3.2. Anaerobic Treatment

The anaerobic treatment experiments of the opium alkaloid wastewaters were carried out in the two stage reactor system over the nine month periods. The experiments discussed in this paper were performed at variable influent COD concentrations in order to investigate the effect of the dilution of the raw wastewater. During the experiments, the samples were analyzed for soluble COD, VFA, pH, TKN, organic loading rate (OLR), sulphate (SO_4^{-2}) and conductivities.

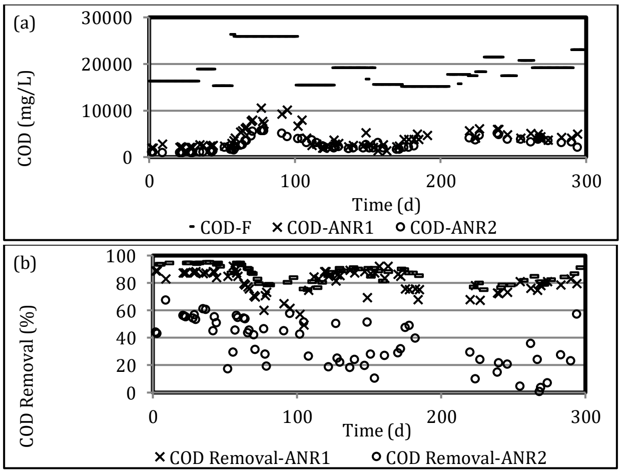
3.2.1. The Effect of Influent COD Concentration During the Anaerobic Digestion

As can be seen from Table 2, the COD concentration of the raw wastewater was about 32,995 ±8811 mg/L. The experiments were performed on the different influent COD values to determine the optimum COD removals during the anaerobic treatment of opium alkaloid wastewaters. The initial COD concentrations of 16120, 15512, and 19000 mg/L were made up by appropriate dilutions. The influent COD concentration of the undiluted wastewater used in the experiments was 25,794 mg/L. Fig.1 a, b, c, d show the COD concentrations, the percentage of COD removals, the VFA concentrations and the organic loading rates (OLR) in both digesters, respectively.

At the influent COD concentrations of 16,120 mg/L, the effluent COD concentrations were about 2041mg/L (87% reduction) in the first reactor (ANR1) and 911 mg/L (55% reduction) in second reactor (ANR2) after a 30 day period. The total COD removal of the two reactors set up was determined as about 94% (Fig.1 a, b).

In order to determine the effect of the influent COD concentrations, the reactors were fed with raw wastewater with 25,794 mg/L COD (Fig.1 a). The effluent COD concentrations were determined as 10,397 mg/L (59% reduction) in ANR1 and 5616mg/L (46% reduction) in ANR2, resulting in the total COD removal was about 78%. The VFA concentration increased up to 4000 mg/L in ANR1 and 2135 mg/L in ANR2 due to the increased influent COD concentration and OLR. In order to reduce free VFA in ANR1 and ANR2, the OLR were lowered from 3.40 kgCOD/ m^{3}day to 1.5 kgCOD/ m^{3}day in the ANR1. Due to this change, the VFA concentration in ANR1 was lowered approximately from 4000 mg/L to 1000 mg/L. Also similar reduction in VFA was achieved in ANR2. The performance of the system was monitored and evaluated by the COD removal and the VFA accumulation (Fig.1 b, c).

Between 154-187th days, when the COD concentration was about 15,512 mg/L, the OLR was increased gradually from 4.4 to 7.27 kgCOD/ m^{3}day in ANR1 (Fig.1 d). The VFA still remained at about 1500 mg/L in ANR1. In the following days, the influent COD concentration was increased up to 21,000 mg/L as a result OLR remained at approximately 8 kgCOD/ m^{3}day at about 60 days in ANR1. (Fig.1 a).



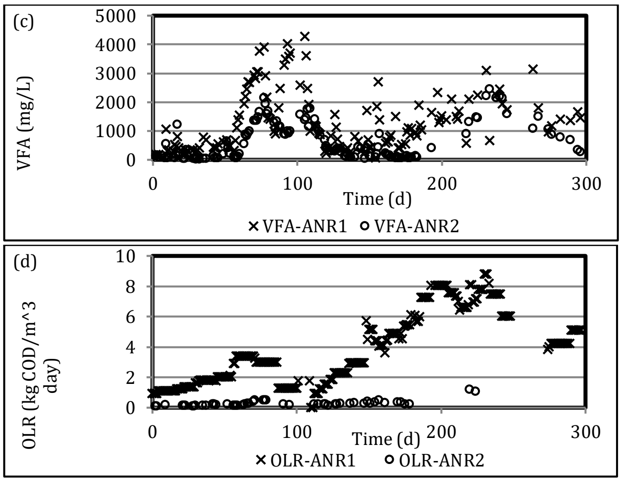


Figure 1. (a) The COD concentrations of the raw wastewater (COD-F) and the effluents from the reactors ANR1 and ANR2, (b) The COD Removals for the reactors (%), (c) The VFA concentration in the raw wastewater (VFA-F) and the effluents from the reactors ANR1 and ANR2, (d) The Organic Loading Rates in the reactors ANR1 and ANR2

3.2.2. The Effect of Conductivity During the Anaerobic Digestion

When the COD concentration and the conductivity were about 19000 mg/L and 20 mS/cm, respectively, the OLR was 7-8 kgCOD/m^{3}day. This is a relatively good result for anaerobic treatment operations. However, in the presence of 25,794 mg/L COD and 30.6 mS/cm conductivity of the raw opium wastewater causes VFA accumulation in the reactors as a result of process imbalance (Fig. 1 c). As the conductivity increased above 20 mS/cm, the inhibition of the organic removal rate was observed. This may be due to the increasing osmotic pressure and possible lower enzymatic activity inside the cell.

The ions present in the influent wastewater ~~as~~ can be seen in Table 4. It should be indicated that Na^+ and K^+ are the most dominant ions. The source of K^+ is known to be the poppy capsules; it is observed that the K^+ concentration in the raw wastewater did not change significantly during the period of this study. However, during the production process, soda is added to poppy capsules, therefore Na^+ concentration is significantly increased. The conductivity of the opium process wastewaters is well correlated primarily with Na^+ and K^+. During the anaerobic treatment experiments, Na^+ and K^+ concentrations were measured as shown in Table 5. The correlation between the total Na^+ and K^+ concentration in the raw wastewater and the conductivity of the wastewater is shown in Fig.2 a. As the total concentration of the Na^+ and K^+ in both influent and effluent of the anaerobic treatment increase, the conductivities increase.

Table 1 Na^+, K^+, and total concentrations and conductivity values of influent and effluent wastewater

	Influent-ANR**1**					Effluent-ANR**2**				
Conductivity (mS/cm)	Na^+	K^+	Na^+	K^+	Na + K	Na^+	K^+	Na^+	K^+	Na + K
	(mg/L)		(mole/L)			(mg/L)		(mole/L)		
17.3	3,107	2,63	0.1351	0.0675	0.2026	2,141	1,903	0.0931	0.0488	0.1419
20.0	3,448	3,17	0.1499	0.0813	0.2312	3,482	2,952	0.1514	0.0757	0.2271
28.6	5,330	5,09	0.2317	0.1305	0.3623	5,330	5,090	0.2317	0.1305	0.3623
30.6	5,746	5,16	0.2498	0.1323	0.3821	5,746	5,158	0.2498	0.1323	0.3821

As can be observed in Fig. 2.a, while the Na^+ and K^+ concentration increase in the wastewater, the conductivity increases as well. It can be concluded that the conductivity and COD-F of the effluents are closely related to the concentrations of Na^+ and K^+.

As shown in Figure 2b, there is a linear relationship between the conductivity and COD-F concentration.

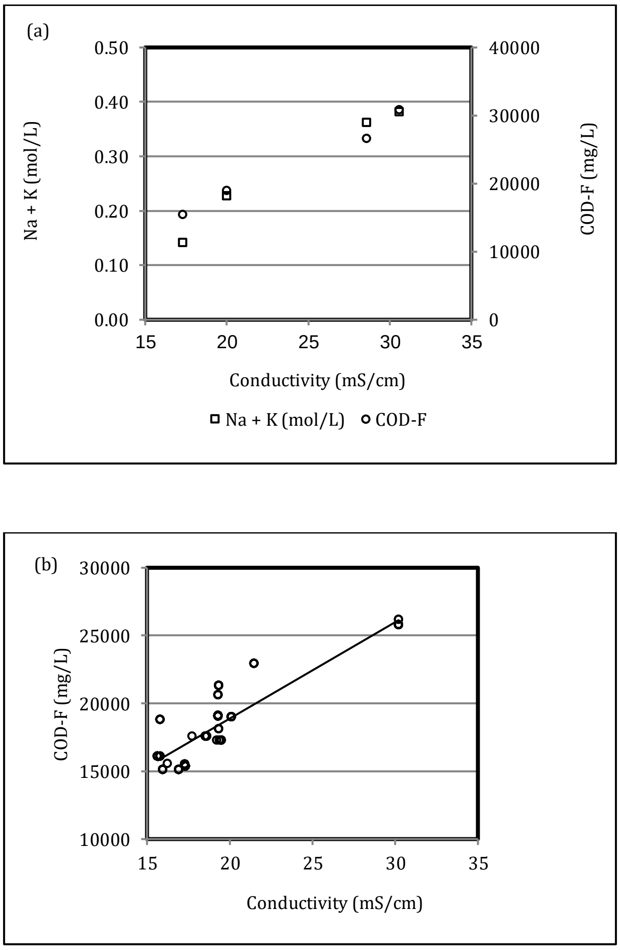
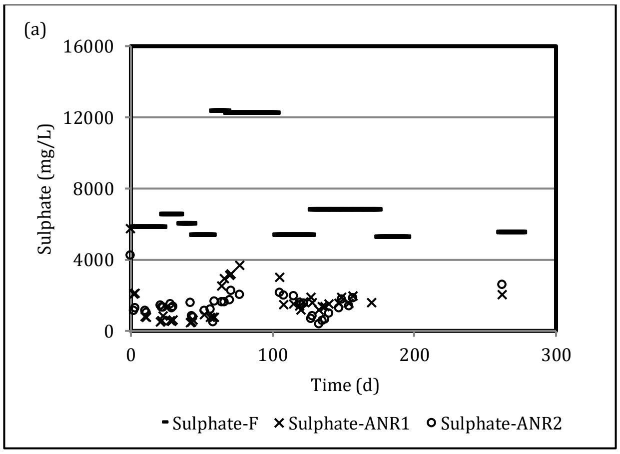


Figure 2. (a)The Relationship between Conductivity, COD-F, and Total concentration of Na^+ and K^+ (b) The relationship between the conductivity and soluble COD concentration.

3.2.3. The Effect of SO_4^{-2} Concentration During the Anaerobic Digestion

In the raw wastewater samples, the SO_4^{-2} concentration was in the range of 11656±4087 mg/L. The SO_4^{-2} concentration was reduced to about 4000 mg/L (%67 reduction) in ANR1 and to about 2000 mg/L (50% reduction) in ANR2. However, when the raw samples were diluted and the initial concentration was reduced to 6500 mg/L, it was observed that the inhibition effect of the SO_4^{-2} was reduced but still significant (Fig. 3 a).



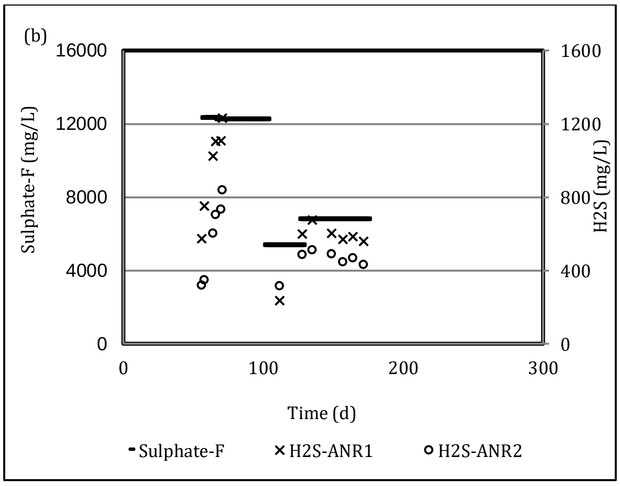


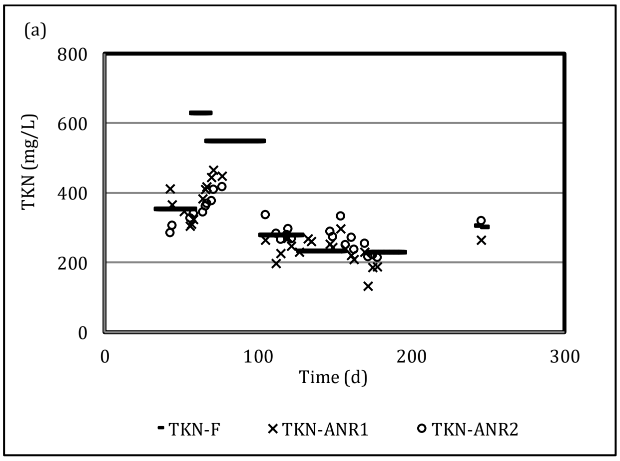
Figure 3. (a)The Sulphate concentrations of the influent wastewater (Sulphate-F) and the effluents of ANR1, and ANR2 (b) The Sulphate concentration of the influent wastewater (Sulphate-F) and the concentrations of H_2S in the reactors ANR1, and ANR2

For the waste streams with a COD/ SO_4^{-2} ratio of over 0.67, there is theoretically enough sulphate present to completely remove the organic matter (COD) available by a sulfidogenic biomass only (Rinzema and Lettinga, 1998). The COD/ SO_4^{-2} ratio was found as 2.88±0.45 in the opium wastewater in this work. Some other industrial wastewaters from molasses-based fermentations, alcohol distilleries, citric acid, fatty acids, leather, paper and board industries produce high COD and SO_4^{-2} concentrations. The COD/ SO_4^{-2} ratios of these wastewaters were reported as 17.9, 15.8, 12, 1, and 54.6, respectively (Polonco and Encia, 2006). As the COD/ SO_4^{-2} value was about 3 in the influent in this work, there was an inhibition effect on the anaerobic treatment but there was still a significant amount of SO_4^{-2} reduction.

It is reported that the inhibition by H_2S concentration above 150 mg/L is possible in anaerobic reactors (Rinzema and Lettinga, 1998). Also, Pol et al. (1998) reported 50 % inhibition at the H_2S concentrations between 50 to 130 mg/L. In this work, H_2S concentration obtained from ANR2 was 447.9 ±64.4 (Fig.3 b) in the case of feeding undiluted wastewaters with high SO_4^{-2} concentration. This measurement does prove the inhibition of SO_4^{-2} reduction during the anaerobic treatment of opium alkaloid wastewaters.

3.2.4. The Effect of TKN Concentration During the Anaerobic Digestion

In the raw wastewater TKN was about 600 mg/L. In anaerobic treatment, it was not reduced in the cases of feeding raw or diluted wastewaters, as can be seen in Fig. 4a. However, by ammonification, TKN was reduced to NH_4 by about 70% and the rest remained as nitrogen bounded to inert COD (Fig. 4b).



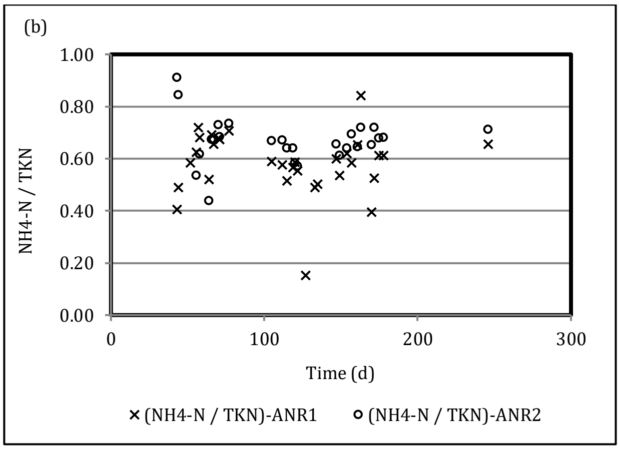
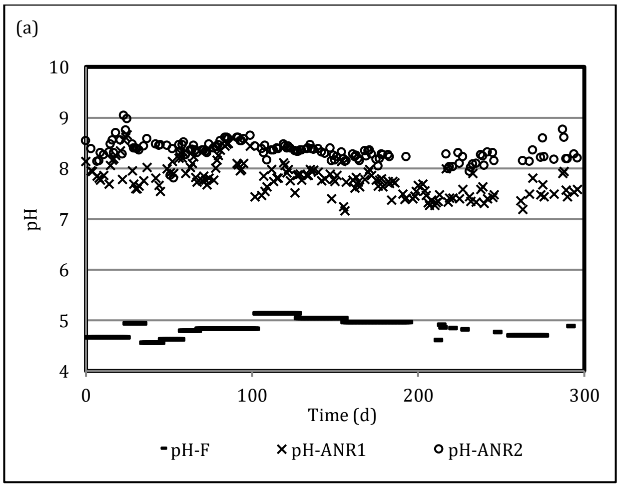


Figure 4. (a) TKN concentration of the feeding wastewater (TKN-F), ANR1, and ANR2 (b) NH4-N/TKN ratio in ANR1 and ANR2

3.2.5. The pH and Alkalinity Values During the Anaerobic Digestion

The pH values observed during the experimental study are shown in Fig. 5 a. pH was not controlled in any operation phases, therefore the pH values of the feed were observed to vary within a range of 4.61-5.14. The increases in pH in the reactors ANR1 and ANR2 during the experiments were oberved as expected.

The alkalinity variations in both reactors are provided in Fig. 5 b. While the organic nitrogen was reduced to NH_4^+, the alkalinity increased as a result of bicarbonate (HCO_3^-) production. In the meantime, the SO_4^{-2} reducing bacteria increased the organic components and reduced SO_4^{-2} to sulphide (S^{-2}). Similarly, HCO_3^- was produced and alkalinity was increased while these reactions occurred. Therefore, pH control was not required during the experiments.



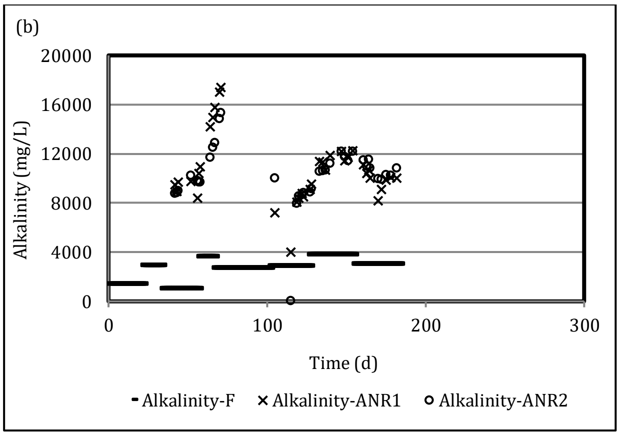


Figure 5. pH (a) and Alkalinity (b) values monitored during the experimental study at the ANR1 and ANR2.

4. Conclusions

The main results of the study of the waste treatment of alkaloid effluents can be summarized as follows:

- COD removal is completed mostly in the first acidogenic reactor.
- During the anaerobic treatment, the presence of 25,794 mg/L COD and 30.2 mS/cm conductivity of the raw opium wastewater causes VFA accumulation in the reactors as a result of process imbalance.
- About 90% of efficiency was observed at the COD concentration around 19,000 mg/L. As the conductivity increased above 20 mS/cm, the inhibition of the organic removal rate was observed due to the increasing osmotic pressure and as a result of possible lower enzymatic activity inside the cell. When the COD concentration and the conductivity were about 20,000 mg/L and 20 mS/cm, respectively, the OLR was 7-8 kgCOD/m^{3}day. This is a relatively good result for anaerobic treatment operations.
- As the COD/ SO_4^{-2} value was about 3 in the influent, there was an inhibition effect on the anaerobic treatment. However, a significant level of SO_4^{-2} reduction was observed. Below the inhibitory level of conductivity (20 mS/cm) COD removal of 90% was achieved even at high OLR levels. Therefore, it can be concluded that the conductivity is more important parameter on the inhibition of anaerobic process compared to SO_4^{-2}.
- The conductivity is more important parameter on the inhibition of anaerobic process compared to SO_4^{-2} concentration.
- pH control was not needed during the experiments because of the sufficient alkalinity

production during the nitrogen reduction to NH4+ and SO_4^{-2} reduction to S^{-2}.

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Conflict of Interest

No conflict of interest was declared by the authors.

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