

Determination of the Biological and Adsorption Removals of Two Phenolic Emerging Micropollutants in a Raw Hospital Wastewater Under Different Sludge Retention Times

Guney G.^{1,*} And Sponza D.T.²

^{1,2} Dokuz Eylul University Engineering Faculty Environmental Engineering Department Tinaztepe Campus 35160 Buca/Izmir/TURKEY

*corresponding author:

e-mail:gokce.pehlivaner@deu.edu.tr, delia.sponza@deu.edu.tr

Abstract

In this study, it was aimed to determine ibuprofen (IBU) and triclosan (TCS) biodegradation and adsorption removals under four different sludge retention times (SRTs) at 5, 30, 45 and 55 days. The amount of adsorption capacity of the micropollutants onto the biological sludge was calculated according to the Langmuir isotherm. Their metabolites which are formed during biological treatment were determined. IBU and TCS biodegradation efficiencies reached to 93.6% and 86.4%, respectively in anaerobic UASB/aerobic CSTR sequential reactor under 55 days of SRT operation. The maximum IBU and TCS adsorption yields were determined as 79.8% and 83.4%, respectively in anaerobic UASB/aerobic CSTR sequential reactor under 55 days of SRT operation. In addition to the evaluation of the IBU and TCS yields, the macropollutants (COD, SCOD and TDS) yields were calculated in the biological reactors. As a result of the study, increasing of the SRT has a positive effect on both macro and micropollutants yields in the anaerobic/aerobic biological reactor system.

Keywords: emerging, hospital wastewater, metabolite, micropollutant, sludge retention time.

1. Introduction

Hospital wastewaters contain a great variety of micropollutants occur in a low concentration range such as ng L^{-1} or $\mu\text{g L}^{-1}$. In Turkey, the micropollutants in the hospital wastewaters are directly discharged into the sewage system without treatment since the conventional sewage/urban treatment plants can only remove macropollutants such as BOD₅, COD, nitrogen and phosphorus. If micropollutants in hospital wastewaters cannot be treated and discharged into the receiving environment, they cause ecotoxic effects in the ecosystem. Some emerging micropollutants are not biologically removed during conventional biological treatment step of the wastewater treatment plants (WWTPs). Moreover, they can be sorbed onto sewage sludge because of their some physicochemical properties. WWTPs are not designed to treat micropollutants efficiently. Hospitals are one of the main sources for the micropollutants discharges to the ecosystem and hospital wastewaters are not treated and directly discharged to the sewage systems in Turkey. In

this study, two emerging micropollutants namely ibuprofen – IBU (non-steroidal anti-inflammatory) and triclosan – TCS (anti-microbial agent) in a raw hospital wastewater were characterized and biologically treated by an anaerobic/aerobic sequential biological reactor system in laboratory conditions. Their metabolites which are formed during biological treatment were determined and their biodegradation (in the supernatant) and adsorption (onto biological sludge) removal mechanisms under four different sludge retention times (SRTs) at 5, 30, 45 and 55 days were investigated. In addition to the evaluation of the IBU, TCS and their metabolites yields, the macropollutants (COD, SCOD and TDS) yields were calculated in the biological reactors. The novelty of the study is to characterize and treat two phenolic emerging micropollutants namely IBU and TCS in a raw hospital wastewater for the first time in Turkey by anaerobic/aerobic sequential biological reactor system in laboratory conditions.

2. Materials and Methods

2.1. Source and characterization of raw hospital wastewater and biomass

Raw hospital wastewater was taken from Dokuz Eylul University Hospital (Izmir, Turkey) sewer channel. Raw hospital wastewater flows through the sewer channel without any treatment and is transferred directly to the wastewater treatment plant. Characterization of the raw hospital wastewater was shown in **Table 1**. Anaerobic and aerobic biomasses were taken from Pakmaya Baker's Yeast Factory's (Izmir, Turkey) anaerobic reactor and aeration tank of the biological treatment process, respectively.

2.2. Reactor configurations and operational conditions of treatment processes

The biological treatability of IBU and TCS in the raw hospital wastewater was investigated using an anaerobic UASB/aerobic CSTR sequential biological reactor system. Their biodegradation removal mechanisms were researched in the supernatants of the biological reactor effluents and their adsorption removal mechanisms were

researched onto the biological sludges of the reactors under four different SRTs at 5, 30, 45 and 55 days.

Table 1. Characterization of the raw hospital wastewater

pH	8.50 ± 0.50
Temperature, (°C)	15.0 ± 5.0
Total nitrogen, (mg L ⁻¹)	1.50 ± 0.50
Total phosphorus, (mg L ⁻¹)	2.00 ± 1.00
Influent COD concentration, (mg L ⁻¹)	1540.00 ± 500.00
Influent SCOD concentration, (mg L ⁻¹)	1000.00 ± 500.00
Influent TDS concentration, (mg L ⁻¹)	196.00 ± 50.00
Influent IBU concentration, (µg L ⁻¹)	7180.00 ± 300.00
Influent TCS concentration, (µg L ⁻¹)	860.00 ± 40.00

Laboratory scale anaerobic/aerobic sequential biological reactor system consists of an upflow anaerobic sludge bed (UASB) reactor without sludge return and an aerobic continuous flow stirred tank (CSTR) reactor without sludge return. The UASB reactor has a total volume of 2 liters. It was equipped with influent and effluent valves, sampling valves and gas outlet valves. Following the UASB reactor without sludge return, an aerobic CSTR reactor has a total volume of 2.5 liters was used. Required temperature conditions for the both reactors were provided with an infrared heater at mesophilic conditions (**Figure 1**). MLVSS values were 3160, 14400 and 17560 mg L⁻¹ at 5 d; 3200, 19120 and 22320 mg L⁻¹ at 30 d; 3560, 20520 and 24080 mg L⁻¹ at 45 d; 3984, 27600 and 31584 mg L⁻¹ at 55 d of SRTs in aerobic, anaerobic and sequential reactors, respectively. The anaerobic UASB/aerobic CSTR sequential reactor system reached steady-state conditions after 48 d of continuous operation which defined with the same effluent COD, SCOD, TDS, IBU and TCS concentrations for consecutive 2 weeks. pH changes, redox potentials, COD, SCOD and TDS yields were also monitored during biological processes.

2.3. Analytical procedure

2.3.1. IBU, TCS and their metabolites measurements in raw hospital wastewater using HPLC

Analytical standard of IBU was purchased from Fluka and its metabolite namely 1-hydroxyibuprofen was purchased from Sigma-Aldrich. Also, analytical standard of TCS was purchased from Fluka and its metabolite namely methyl triclosan was purchased from Sigma-Aldrich. Solutions of IBU, 1-hydroxyibuprofen, TCS and methyl triclosan were diluted by using methanol and was stirred for 2 hours on magnetic stirrers and was ensured a homogenous distribution. HPLC analysis were performed on an Agilent 1100 Series HPLC equipped with a diode array detector. IBU and 1-hydroxyibuprofen were measured according to Hassan *et al.* (Hassan *et al.*, 2008). The retention times for IBU and 1-hydroxyibuprofen were calculated as 5.778 and 4.750 min, respectively. TCS and methyl triclosan were measured according to Maarroof and Uysal (Maarroof and

Uysal, 2014). The retention times for TCS and methyl triclosan were calculated as 3.606 and 2.550 min, respectively. Regression coefficients (R²) of the calibration curves were above 0.999 for IBU, 1-hydroxyibuprofen, TCS and methyl triclosan.

2.3.2. Extractions of IBU, TCS and their metabolites from raw hospital wastewater

IBU, TCS and their metabolites (1-hydroxyibuprofen and methyl triclosan) were extracted from raw hospital wastewater by solid-phase extraction (SPE) method. Oasis HLB (200 mg) Cartridges were used in the experiments and the experiments were carried out according to Diwan *et al.* (Diwan *et al.*, 2010).

2.3.3. Adsorption and biodegradation tests of IBU and TCS

For the adsorption tests, quartz glass reactors with a volume of 250 mL were used and they were filled with 10 mL activated aerobic and anaerobic granule sludges autoclaved at 130 °C at 1.8 atmosphere pressure for 45 min, separately. Then, they were agitated for 24 h using a shaker at 25°C. After the agitation, each reactor was centrifuged for 5 min, then the supernatant was removed and extracted using the afore-mentioned solid-phase extraction method. For the biodegradation tests, the supernatants were taken from the anaerobic UASB and aerobic CSTR reactors and extracted using the afore-mentioned solid-phase extraction method.

2.4. Other measurement methods

Temperature was measured with a thermometer. pH, dissolved oxygen (DO) concentrations and oxidation reduction potentials (ORPs) were measured with WTW probes. Total nitrogen and total phosphorus were measured with reagent kits in a Photometer Nova 60/Spectroquant. The MLVSS, COD, SCOD and TDS were measured based on Standard Methods (Standard Methods, 1992).

3. Results and Discussion

3.1. COD, SCOD and TDS removals in anaerobic UASB/aerobic CSTR sequential biological reactor system

The temperatures of the both anaerobic UASB and aerobic CSTR reactors were kept constant at 35±2 °C under mesophilic conditions. pH values were kept constant at 8.50 for anaerobic UASB and 7.50 for aerobic CSTR reactors by adding NaOH. DO concentrations and ORPs were stabilized 0.00 mg L⁻¹ and -600 mV for anaerobic UASB and 2.00 mg L⁻¹ and +200 mV for aerobic CSTR reactors, respectively. Higher COD yields were obtained at 55 days of SRT (94.1%) than 45 (92.2%), 30 (89.7%) and 5 (87.8%) days of SRTs in anaerobic/aerobic sequential reactor system. Moreover, higher SCOD yields were obtained at 55 days of SRT (91.1%) than 45 (88.1%), 30 (84.4%) and 5 (82.0%) days of SRTs in anaerobic/aerobic sequential reactor system. Higher TDS yields were obtained at 55 days of SRT (65.8%) than 45 (64.2%), 30 (55.1%) and 5 (36.2%) days of SRTs in anaerobic/aerobic sequential reactor system. As expected, increasing SRT increased the COD and TDS removal efficiencies in the biological treatment (Table 2).

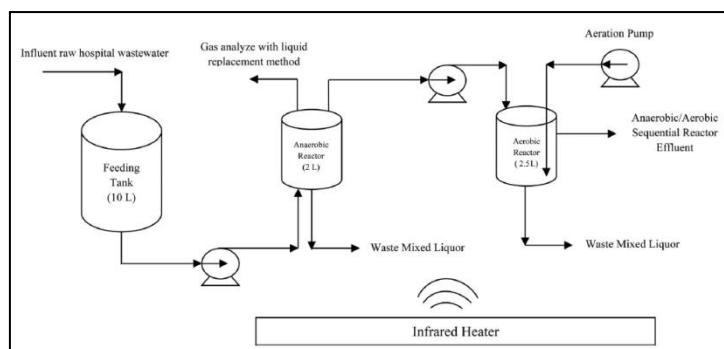


Figure 1. Lab. scale anaerobic UASB/aerobic CSTR sequential biological reactor system

Table 2. Influent and effluent COD, SCOD, TDS concentrations and yields in anaerobic UASB/aerobic CSTR sequential biological reactor system

SRT (d)	MLVSS (mg L ⁻¹)	Chemical Oxygen Demand (COD)			Soluble Chemical Oxygen Demand (SCOD)			Total Dissolved Solids (TDS)		
		Inf.	Eff.	Yield	Inf.	Eff.	Yield	Inf.	Eff.	Yield
		(mg L ⁻¹)	(mg L ⁻¹)	(%)	(mg L ⁻¹)	(mg L ⁻¹)	(%)	(mg L ⁻¹)	(mg L ⁻¹)	(%)
5	17560	1540.00	187.88	87.8	1000.00	180.00	82.0	196.00	124.85	36.3
30	22320	1540.00	158.62	89.7	1000.00	156.00	84.4	196.00	88.40	54.9
45	24080	1540.00	120.12	92.2	1000.00	119.00	88.1	196.00	70.17	64.2
55	31584	1540.0000	90.26	94.1	1000.00	89.00	91.1	196.00	67.03	65.8

3.2. Adsorption yields of IBU and TCS in the anaerobic UASB/aerobic CSTR sequential biological reactors

Adsorption removal efficiencies of IBU and TCS onto the biological sludges of the anaerobic UASB and aerobic CSTR reactors were researched in the eluates of the sludges of the biological reactors. The maximum IBU adsorption yields on the sludge of the anaerobic reactor were obtained as 12.2%, 29.5%, 35.0% and 37.5% with an IBU influent of 7180.00 $\mu\text{g L}^{-1}$ and effluent IBU concentrations of 6304.04, 5061.90, 4667.00 and 4487.50 $\mu\text{g L}^{-1}$ at 5, 30, 45 and 55 days of SRTs, respectively (Table 3). Following the anaerobic reactor, the IBU removal in the sludge of the aerobic reactor reached 14.0%, 32.6%, 37.3% and 39.4% with influent IBU concentrations of 3619.87, 2958.30, 2716.98 and 2387.25 $\mu\text{g L}^{-1}$ and effluent IBU concentrations of 3113.09, 1993.89, 1703.55 and 1446.67 $\mu\text{g L}^{-1}$ at 5, 30, 45 and 55 days of SRTs, respectively (Table 3). Both anaerobic and aerobic reactors did not provide sufficient IBU removal on the sludge. It was found that IBU removal with adsorption onto sludge was found to be not significant. The sequential anaerobic UASB/aerobic CSTR reactor system exhibited only 56.6%, 72.2%, 76.3% and 79.8% total IBU removals on sludge at 5, 30, 45 and 55 days of SRTs with an IBU influent of 7180.00 $\mu\text{g L}^{-1}$ and effluent IBU concentrations of 3113.09, 1993.89, 1703.55 and 1446.67 $\mu\text{g L}^{-1}$, respectively (Table 3). The maximum TCS adsorption yields on the sludge of the anaerobic reactor were obtained as 24.2%, 46.4%, 48.7% and 51.3% with a TCS influent of 860.00 $\mu\text{g L}^{-1}$ and with effluent TCS concentrations of 651.88, 460.96, 441.18 and 418.82 $\mu\text{g L}^{-1}$ at 5, 30, 45 and 55 days of SRTs, respectively (Table 3). Following the anaerobic reactor, TCS removals in aerobic reactor reached

28.0%, 50.3%, 56.0% and 59.2% with influent TCS concentrations of 642.88, 376.34, 358.38 and 350.58 $\mu\text{g L}^{-1}$ and effluent TCS concentrations of 462.87, 187.04, 157.69 and 143.04 $\mu\text{g L}^{-1}$ at 5, 30, 45 and 55 days of SRTs, respectively (Table 3). Both anaerobic and aerobic reactors did not provide sufficient TCS removal via adsorption on the sludge. However, TCS removal via adsorption in the sequential reactor system reached 46.2%, 78.2%, 81.7% and 83.4% TCS removals with a TCS influent of 860.00 $\mu\text{g L}^{-1}$ and effluent TCS concentrations of 462.87, 187.04, 157.69 and 143.04 $\mu\text{g L}^{-1}$ after 5, 30, 45 and 55 days of SRTs, respectively (Table 3). The adsorption kinetics of IBU and TCS could be explained using Langmuir isotherm. The amount of adsorption capacity of anaerobic sludge was calculated for IBU as 26.42 $\mu\text{g g}^{-1}$ ($q_{e,\text{anaerobic,IBU}} = 26.42 \mu\text{g g}^{-1}$, $R^2 = 0.99$) and for TCS as 32.37 $\mu\text{g g}^{-1}$ ($q_{e,\text{anaerobic,TCS}} = 32.37 \mu\text{g g}^{-1}$, $R^2 = 0.99$) (Data not shown). The amount of adsorption capacity of aerobic sludge was calculated for IBU as 28.74 $\mu\text{g g}^{-1}$ ($q_{e,\text{aerobic,IBU}} = 28.74 \mu\text{g g}^{-1}$, $R^2 = 0.99$) and for TCS as 34.25 $\mu\text{g g}^{-1}$ ($q_{e,\text{aerobic,TCS}} = 34.25 \mu\text{g g}^{-1}$, $R^2 = 0.99$) (Data not shown).

3.3. Biodegradation yields in the anaerobic UASB/aerobic CSTR sequential biological reactor effluents for IBU, TCS and their metabolites

Biodegradation removal efficiencies were researched in both anaerobic UASB and aerobic CSTR reactors. During the 5, 30, 45 and 55 days of SRT operation, with an influent IBU concentration of 7180.00 $\mu\text{g L}^{-1}$ the maximum IBU removal efficiencies in anaerobic reactors were obtained as 45.2%, 56.4%, 60.5% and 65.7% with effluent IBU concentrations of 3934.64, 3130.48, 2836.10 and 2461.08 $\mu\text{g L}^{-1}$, respectively (Table 3). 1-hydroxyibuprofen a metabolite of IBU was measured after

Table 3. Adsorption and biodegradation yields for IBU, TCS and formations of their metabolites

Micropollutant	SRT (d)	Anaerobic Reactor			Aerobic Reactor			Sequential Reactor		
		Inf. (µg L ⁻¹)	Eff. (µg L ⁻¹)	Yield (%)	Inf. (µg L ⁻¹)	Eff. (µg L ⁻¹)	Yield (%)	Inf. (µg L ⁻¹)	Eff. (µg L ⁻¹)	Yield (%)
IBU (Adsorption)	5	7180.00	6304.04	12.2	3619.87	3113.09	14.0	7180.00	3113.09	56.6
	30	7180.00	5061.90	29.5	2958.30	1993.89	32.6	7180.00	1993.89	72.2
	45	7180.00	4667.00	35.0	2716.98	1703.55	37.3	7180.00	1703.55	76.3
	55	7180.00	4487.50	37.5	2387.25	1446.67	39.4	7180.00	1446.67	79.8
IBU (Biodegradation)	5	7180.00	3934.64	45.2	3619.87	1625.32	55.1	7180.00	1422.15	80.2
	30	7180.00	3130.48	56.4	2958.30	967.36	67.3	7180.00	893.84	87.6
	45	7180.00	2836.10	60.5	2716.98	679.25	75.0	7180.00	635.10	91.2
	55	7180.00	2461.08	65.7	2387.25	487.00	79.6	7180.00	461.68	93.6
1-Hydroxyibuprofen	SRT (d)	Inf. (µg L ⁻¹)	Eff. (µg L ⁻¹)	Formation (%)	Inf. (µg L ⁻¹)	Eff. (µg L ⁻¹)	Formation (%)	-	-	-
	5	3934.64	314.77	8.0	1625.32	203.17	12.5	-	-	-
	30	3130.48	172.18	5.5	967.36	73.52	7.6	-	-	-
	45	2836.10	119.12	4.2	679.25	44.15	6.5	-	-	-
55	2461.08	73.83	3.0	487.00	25.32	5.2	-	-	-	

Micropollutant	SRT (d)	Anaerobic Reactor			Aerobic Reactor			Sequential Reactor		
		Inf. (µg L ⁻¹)	Eff. (µg L ⁻¹)	Yield (%)	Inf. (µg L ⁻¹)	Eff. (µg L ⁻¹)	Yield (%)	Inf. (µg L ⁻¹)	Eff. (µg L ⁻¹)	Yield (%)
TCS (Adsorption)	5	860.00	651.88	24.2	642.88	462.87	28.0	860.00	462.87	46.2
	30	860.00	460.96	46.4	376.34	187.04	50.3	860.00	187.04	78.2
	45	860.00	441.18	48.7	358.38	157.69	56.0	860.00	157.69	81.7
	55	860.00	418.82	51.3	350.58	143.04	59.2	860.00	143.04	83.4
TCS (Biodegradation)	5	860.00	710.36	17.4	642.88	516.88	19.6	860.00	452.27	47.4
	30	860.00	398.66	53.6	376.34	163.33	56.6	860.00	152.55	82.3
	45	860.00	375.27	56.4	358.38	134.39	62.5	860.00	126.33	85.3
	55	860.00	362.17	57.9	350.58	122.70	65.0	860.00	116.56	86.4
Methyl triclosan	SRT (d)	Inf. (µg L ⁻¹)	Eff. (µg L ⁻¹)	Formation (%)	Inf. (µg L ⁻¹)	Eff. (µg L ⁻¹)	Formation (%)	-	-	-
	5	710.36	67.48	9.5	516.88	64.61	12.5	-	-	-
	30	398.66	22.32	5.6	163.33	10.78	6.6	-	-	-
	45	375.27	16.89	4.5	134.39	8.06	6.0	-	-	-
55	362.17	11.59	3.2	122.70	6.14	5.0	-	-	-	

the anaerobic biological treatment. During the 5, 30, 45 and 55 days of SRT operation, 1-hydroxyibuprofen formations were obtained as 314.77, 172.18, 119.12 and 73.83 $\mu\text{g L}^{-1}$, respectively (Table 3). Following the anaerobic reactor, during the 5, 30, 45 and 55 days of SRT operation, the IBU yields reached 55.1%, 67.3%, 75.0% and 79.6% in the aerobic reactor with influent IBU concentrations of 3619.87, 2958.30, 2716.98 and 2387.25 $\mu\text{g L}^{-1}$ and with the effluents of 1625.32, 967.36, 679.25 and 487.00 $\mu\text{g L}^{-1}$, respectively (Table 3). During the 5, 30, 45 and 55 days of SRT operation, 1-hydroxyibuprofen formations were obtained as 203.17, 73.52, 44.15 and 25.32 $\mu\text{g L}^{-1}$, respectively (Table 3). The anaerobic reactor did not provide sufficient IBU removals, however, aerobic reactor enhanced the IBU removals in the supernatant. The sequential reactor system obtained 80.2 %, 87.6%, 91.2% and 93.6% IBU removals with an IBU influent of 7180.00 $\mu\text{g L}^{-1}$ and the effluents of 1422.15, 893.84, 635.10 and 461.68 $\mu\text{g L}^{-1}$, under 5, 30, 45 and 55 days of SRT operation, respectively (Table 3). During 5, 30, 45 and 55 days of SRT operation, with a TCS influent of 860.00 $\mu\text{g L}^{-1}$ the maximum TCS removal efficiencies in the anaerobic reactors were obtained as 17.4%, 53.6%, 56.4% and 57.9% with the effluents of 710.36, 398.66, 375.27 and 362.17 $\mu\text{g L}^{-1}$, respectively (Table 3). Methyl triclosan a metabolite of TCS was measured after the anaerobic biological treatment. During the 5, 30, 45 and 55 days of SRT operation, methyl triclosan formations were obtained as 67.48, 22.32, 16.89 and 11.59 $\mu\text{g L}^{-1}$, respectively (Table 3). Following the anaerobic reactor, during the 5, 30, 45 and 55 days of SRT operation, the TCS yields reached 19.6%, 56.6%, 62.5% and 65.0% in the aerobic reactor with TCS influent concentrations of 642.88, 376.34, 358.38 and 350.58 $\mu\text{g L}^{-1}$ and the effluent TCS concentrations of 516.88, 163.33, 134.39 and 122.70 $\mu\text{g L}^{-1}$, respectively (Table 3). During the 5, 30, 45 and 55 days of SRT operation, methyl triclosan formations were obtained as 64.61, 10.78, 8.06 and 6.14 $\mu\text{g L}^{-1}$, respectively (Table 3). The anaerobic reactor did not provide sufficient TCS removals, but, aerobic reactor enhanced the TCS removals in the supernatant. The sequential reactor system obtained 47.4%, 82.3%, 85.3% and 86.4% TCS removals with a TCS influent of 860.00 $\mu\text{g L}^{-1}$ and the effluents of 452.27, 152.55, 126.33 and 116.56 $\mu\text{g L}^{-1}$, after 5, 30, 45 and 55 days of SRT operation, respectively (Table 3).

4. Conclusions

The biodegradation and adsorption yields for IBU and TCS were insufficient both for anaerobic UASB and aerobic CSTR biological reactors at short (5 days) SRT operations. However, increasing the SRT from 5 days to 30 and 45 days enhanced both the biodegradation and adsorption removal efficiencies for IBU and TCS. Increasing the SRT in the anaerobic UASB/aerobic CSTR sequential biological reactor had a significant effect on the removals of IBU and TCS from the raw hospital wastewater and affected positively the removal performances of biological reactors. The maximum IBU biodegradation efficiency reached to 93.6% in anaerobic UASB/aerobic CSTR sequential reactor under 55 days of SRT operation. It can be said that increasing SRT from 5 days to 55 days enhanced IBU yield in the supernatant. The maximum IBU yield removed by adsorption was determined as 79.8% in anaerobic UASB/aerobic CSTR sequential reactor under

55 days of SRT operation. Adsorption yield was increased by increasing the SRT on the biological sludges. The maximum TCS biodegradation efficiency reached to 86.4% in anaerobic UASB/aerobic CSTR sequential reactor under 55 days of SRT operation. It can be said that increasing SRT from 5 days to 55 days enhanced TCS yield in the supernatant. The maximum TCS adsorption yield was determined as 83.4% in anaerobic UASB/aerobic CSTR sequential reactor under 55 days of SRT operation. Increasing the SRT had a positive effect on the adsorption yield of TCS. Falås *et al.* and Ferrando-Climent *et al.* reported that increasing SRT and increasing MLVSS concentrations had a positive effect on the micropollutants' removal in biological treatment systems (Falås *et al.*, 2012 and Ferrando-Climent *et al.*, 2012). The results obtained in this study are compatible with this explanation. An increase in SRT and MLVSS was increased the both macro (COD, SCOD and TDS) and micropollutants (IBU, 1-hydroxyibuprofen, TCS and methyl triclosan) yields in anaerobic UASB/aerobic CSTR sequential biological reactor system.

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