Semra ILHAN<sup>2</sup>

# **Treatment of Cake Production Wastewater in Upflow Anaerobic Packed Bed Reactors**

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## Abstract

The results presented in this paper are from studies on a laboratory-scale upflow anaerobic packed bed (UAPB) reactors treating cake production wastewater at increasing organic loading rates of 1.36-6.48 g COD l<sup>-1</sup>day<sup>-1</sup>. The hydraulic retention times (HRT) of the reactors ranged from 120-26 hours for reactors during the 115 days of the experiment. At 5.88 g COD l<sup>-1</sup> per day<sup>-1</sup> organic load and 29 hours retention time, a 98-99 % total COD removal was achieved in two reactors. The gas production increased with a rising organic loading rate of up to 9.28 l CH<sub>4</sub> l<sup>-1</sup> day<sup>-1</sup> in the first-stage reactor and 0.73 l CH<sub>4</sub> l<sup>-1</sup> day<sup>-1</sup> in the second-stage reactor. The study demonstrated the suitability of UAPB reactors for treatment of cake production wastewater.

Keywords: Anaerobic packed-bed reactor; cake production wastewater; methane production; COD removal; organic load.

## **INTRODUCTION**

The use of anaerobic digestion as a means of treating waste products has increased in the past 30 years. The process involves the treatment of agricultural and industrial wastes of varying types, with a resulting end production of biogas. Interest in the anaerobic treatment of agro-industry wastes is increasing due to its being both an economical, and ecologically sound approach, as well as having lower energy requirements, these being just a few advantages among several others, when compared with aerobic treatment processes [1,2].

Anaerobic digestion is a complex, natural, multi-stage process. During the process, organic compounds are degraded through a variety of intermediates into methane and carbon dioxide, by the activity of a consortium of microorganisms. Interdependence of the bacteria is a key factor in the anaerobic digestion process. Instability during both the startup and operation of the anaerobic degradation process can be problematic due to the low specific growth rate of the methanogenic microorganisms involved [1].

Wastewaters produced in the food industry are characterized by their high organic content, most of which is being composed of easily biodegradable compounds such as carbohydrates, proteins and in some cases, smaller contents of lipids [3]. Food processing wastewaters are suitable for anaerobic treatment processes because they rarely contain toxicants or inhibitory compounds in their composition [4]. The use of packed-bed reactors to treat different kinds of wastewater has also been previously reported, with two such examples being, dairy and brewery wastewaters [1,4].

In this study both the anaerobic treatability and methane generation potential of cake production wastewater were investigated an upflow anaerobic packed bed (UAPB) reactor with plastic balls as support material. For the purpose of the UAPBR experiments, operational parameters such as hydraulic retention time (HRT) and loading rate were investigated.

## MATERIAL AND METHOD

#### **Characterization of wastewaters**

Cake production wastewaters (CPW) were obtained from a cake production factory located near the city of Eskisehir, Turkey. Samples collected were stored at 4°C during the study. Table 1 shows the typical characteristic parameters of the wastewaters.

Table 1. Characterization of the CPW

Parameters	CPW
pH	12.25
Chemical oxygen demand (COD)(mg l-1)	12000±200
Total solids (TS) (mg l <sup>-1</sup> )	$11800 \pm 250$
Suspended solids (SS) (mg l <sup>-1</sup> )	6250±150
Volatile suspended solids (VSS)(mg l <sup>-1</sup> )	3300±200
Total kjeldahl nitrogen (TKN)(mg l-1)	44.8±0,8
Total phosphorus (TP) (mg l <sup>-1</sup> )	58.8±0,8

#### Upflow Anaerobic Packed Bed (UAPB) Reactor

This laboratory scale study was performed using two reactor designs (Fig 1). Each of the two reactors had a total liquid volume of 5.0 l and was filled up to 4.3 l of their volume with plastic balls. A rough surface was achieved in order to retain the bacterial biomass. The temperature of the reactors was maintained at mesophilic conditions (37 °C) through the use of electrical heating mats. The feed was then introduced through the bottom of each reactor by means of peristaltic pumps.

#### Seed sludge

A mixed mesophilic sludge (suspended solids 60 g l<sup>-1</sup> and volatile suspended solids 38 g l<sup>-1</sup>) obtained from anaerobic

digesters at the Ankara Municipal Wastewater Treatment Plant, Turkey, was used as the inoculum in the UAPB reactors.



Figure 1. Scheme of upflow anaerobic packed bed reactor used for anaerobic treatment of CPW

#### **Basal medium**

The composition of the basal medium (BM) used in the experiments was as follows (concentrations of the constituents are given in parentheses as mg l<sup>-1</sup>): NH<sub>4</sub>Cl (1200), MgSO<sub>4</sub>, 7H<sub>2</sub>O (400), KCl (400), Na<sub>2</sub>S.9H<sub>2</sub>O (300), CaCl<sub>2</sub>.2H<sub>2</sub>O (50), (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> (80), FeCl<sub>2</sub>.4H<sub>2</sub>O (40), CoCl<sub>2</sub>.6H<sub>2</sub>O (10), KI (10), MnCl<sub>2</sub>.4H<sub>2</sub>O (0.5), CuCl<sub>2</sub>.2H<sub>2</sub>O (0.5), ZnCl<sub>2</sub> (0.5), AlCl<sub>3</sub>.6H<sub>2</sub>O (0.5), NaMOO<sub>4</sub>.2H<sub>2</sub>O (0.5), H<sub>3</sub>BO<sub>3</sub> (0.5), NiCl<sub>2</sub>.6H<sub>2</sub>O (0.5), NaWO<sub>4</sub>.2H<sub>2</sub>O (0.5), Na<sub>2</sub>SeO<sub>3</sub> (0.5), cysteine (10). This basal medium contained all of the micro and macro nutrients required for an optimum anaerobic microbial growth [5,6].

#### **Operational conditions**

In previous experiments, biochemical methane potential (BMP) experiments were performed in order to determine the anaerobic biodegradability and biogas production of the cake production wastewater (CPW) under investigation. In order to compare the supplementation of nutrient and trace metals in gas production, one out of two sets of serum bottles were given basal medium (BM) for wastewater. According to the BMP results, higher COD removal and the methane generation was observable with an initial COD concentration of 12000 mg l<sup>-1</sup> in the biomass with BM [7].

In order to achieve the nutrient and trace metal concentrations given in the BM for the influent of the UAPB reactor, the wastewater described in Table 1 was diluted by a ratio of 4/5 with BM. This mixture was then used as the feed solution in the experiments. In other words, each liter of feed solution consisted of 0.8 l of the cake production wastewater described in Table 1 and 0.2 l of BM. Thus, feed solution had an average COD of 7000±200 mg l<sup>-1</sup>. The pH of the feed solution was adjusted to 7.05±0.05 by the addition of 1M HCl. The organic loading rate (OLR) applied to the first-stage reactor ranged from 1.4-6.48 g COD l<sup>-1</sup>day<sup>-1</sup>, corresponding to a hydraulic retention time (HRT) of 120-26 hour. The organic loading rate (OLR) applied to the second-stage reactor ranged from 0.42-0.85 g COD l<sup>-1</sup>day<sup>-1</sup> corresponding to an HRT of 74-26 hour.

## Analytical methods

pH measurements were obtained with a pH meter (WTW, Inolab Level 2) and a pH probe (BO11207-023, WTW). COD,

total solids (TS), suspended solids (SS), volatile suspended solids (VSS), alkalinity, volatile fatty acids (VFA) and total kjeldahl nitrogen were all measured by following standard methods, 5220B, 2540B, 2540D, 2540E, 2320B, 5560C, 4500- $N_{org}$ B, respectively [8].

Total phosphorus was measured with the Merck cell test (1.14729.0001) and Cecil 4002 UV visible spectrophotometer (England).

The volume of methane produced was determined daily by the liquid displacement method, after first removing  $CO_2$  by adsorption into NaOH solution [6,9].

#### Statistical analysis

Regression analysis between HRT and COD removal efficiency was performed using the SPSS 15.0. The linear correlation was assessed with  $r^2$ . The  $r^2$  value is the correlation coefficient and reflects statistical significance between dependent and independent variables [10]. Analysis of variance (ANOVA) test was used to assess the data obtained in reactors using SPSS 15.0.

## **RESULTS AND DISCUSSION**

Figure 2 and 3 summarize the HRT, applied organic loading rates, influent and effluent total COD concentration, pH, bicarbonate alkalinity values and effluent VFA concentrations. As can be seen in Table 2, COD removal increased from 63-93% during the first-reactor stage, while OLR increased from 1.4 to 5.88 g COD each day and HRT decreased from 120-29 hours. At an OLR of 5.88-6.48 g COD per day, a decrease in efficiency was observed (87  $\pm$ 1%). Effluent from the first-stage of the UAPB reactor was fed to the second-stage UAPB reactor. When considering the performance of the second-stage UAPB reactor, a total COD removal efficiency of 46-84 % was demonstrated, an effluent quality which can be directly discharged into a receiving environment. In a prior study conducted by Parawira et al. (2005), a laboratory-scale upflow sludge blanked (UASB) reactor and an upflow anaerobic packed- bed (UAPB) reactor treating potato leachate were investigated. The potato leachate was treated at increasing organic loading rates from 1.5 to 7.0 g COD 1<sup>-1</sup> day<sup>-1</sup>. The maximum organic loading rates possible in the APB reactors for stable operation were approximately 4.7 g COD l-1day-1.

The variations of HRT and COD removal of UAPB reactors are shown in Table 2 and 3. The regression analysis indicated that the linear relationship between HRT and COD removal efficiency was statistically significant in first-stage reactor ( $r^2$ =0.71, P=0.00, F=26.52). As the HRT values decreased the COD removal increased. In second-stage reactor, significant quadratic correlation between HRT and COD removal efficiency was observed ( $r^2$ =0.81, P=0.03, F=14.67).

The pH in both of the reactors remained more or less constant for all of the HRT studied, with 7.2 and 7.9 as extreme values (Fig 2d and 3d). This stability can be attributed to carbonate/bicarbonate buffering. When the level of alkalinity is above 1000 mg l<sup>-1</sup> the system has sufficient buffer capacity. The alkalinity of the original wastewater was very low; therefore 2 g l<sup>-1</sup> NaHCO<sub>3</sub> was added to it. The buffering guards against

possible acidification of the reactor giving a pH of the same level that is equally optimal for methanogenic bacteria. The high pH values and the buffering capacity are a guarantee as opposed to an acidification of the reactor that could be caused by a sudden overloading of the reactor [11]. The VFA/alkalinity ratio can be used as a measure of process stability; when this ratio is less than 0.3-0.4 the process it is considered to be operating favorably without risk of acidification [11]. As was discernible (Table 2 and 3), the ratio values were lower than the suggested limit value in all HRT studies.



**Figure 2**. Anaerobic digestion of cake production wastewater in the first-stage upflow anaerobic packed bed reactor:(a) HRT; (b) OLR; (c)influent and effluent COD concentrations; (d) pH; (e) influent and effluent bicarbonate alkalinity; (f) effluent VFA (acetic acid) concentration

The values of volumetric methane production rates for each OLR studied in the first-stage UAPB reactor and second-stage UAPB reactor are also given in Table 2 and 3. The methane yields in the first-stage UAPB reactor increased linearly with increasing OLR up to 9.28 1 CH<sub>4</sub> 1<sup>-1</sup> day<sup>-1</sup> at 5.88 g COD 1<sup>-1</sup>

day<sup>-1</sup>. When the OLR was increased to 6.48 g COD l<sup>-1</sup> day<sup>-1</sup> in the first-stage UAPB reactor a decrease in methane production and escalating concentration of VFA was observed (Table 2 and Fig 2f). Methane yield seems to be a representative parameter for process monitoring. Many authors have determined the



**Figure 3.** Anaerobic digestion of cake production wastewater in the second-stage upflow anaerobic packed bed reactor:(a) HRT; (b) OLR; (c)influent and effluent COD concentrations; (d) pH; (e) influent and effluent bicarbonate alkalinity; (f) influent and effluent VFA ( as acetic acid) concentration

methane yield of biofilm reactors under steady-state conditions, and reported different results depending on the substrate and the operating conditions [1,12]. Kalyuzhnyi et al (1998) obtained a methane yield of 0.3 1 CH<sub>4</sub>g<sup>-1</sup> COD day<sup>-1</sup> at an OLR of 6 g COD l<sup>-1</sup> day<sup>-1</sup> in an UASB reactor treating potato-maize wastewater. Parawira et al. (2005) reported that the methane yields of an UASB reactor and an UAPB reactor were below the expected values of 0.35 1 CH<sub>4</sub>g<sup>-1</sup> COD day<sup>-1</sup>. In this study, the methane yields of both reactors were above expected values, since more or less steady state conditions were achieved under these experimental conditions.

When the performance of the two-stage UAPB reactor is considered, total COD removal efficiency was seen at 98-99% and effluent COD concentration was below the discharge limits.

## CONCLUSION

When considered as a whole, this study indicated that the high rate anaerobic treatment of cake production wastewater in

packed bed reactors is a very efficient method. Furthermore, the following conclusions can be drawn based on the experimental results of this study.

- 1. HRT values as low as 29-36 hour can be used for the anaerobic treatment of cake production wastewater, with a COD removal efficiency of 89-93 % at influent COD concentration of 7000±200 mg l<sup>-1</sup>.
- 2. A second-stage UAPB reactor achieved an additional COD removal efficiency of 46-84 %.
- 3. Nutrient, trace metal and alkalinity supplementation is vital for the anaerobic treatment of cake production wastewaters.

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Days	HRT	Influent parameters						Effluent parameters							
		COD	SS	OLR	рН	Alkalinity	COD	SS	Gas Production	рН	Alkalinity	VFA	COD Removal (%)	VFA/ Alkalinity	
0-14	120	7000	4500	1.4	7.1	2000	2600	450	1.49	7.2	1950	220	63	0,11	
15-18	74	7000	4500	2.28	7.05	2000	2300	410	2.58	7.3	2000	225	67	0,11	
19-22	60	7000	4500	2.8	7	2000	1350	400	3.83	7.2	2100	240	80	0,12	
23-27	53	7000	4500	3.18	7.1	2000	2350	430	3.58	7.2	1850	220	66	0,10	
28-37	60	7000	4500	2.8	7.1	2000	1300	410	3.88	7.2	1800	225	81	0,12	
38-47	53	7000	4500	3.18	7.1	2000	1200	410	4.46	7.3	2050	200	82	0,11	
48-57	48	7000	4500	3.5	7	2000	950	395	5.14	7.2	2200	220	86	0,10	
58-65	44	7000	4500	3.77	7.1	2000	900	390	5.56	7.3	2250	220	87	0,10	
66-75	36	7000	4500	4.67	7.13	2000	750	380	7.06	7.5	2360	224	89	0,09	
76-86	31	7000	4500	5.38	7.1	2000	700	380	8.21	7.5	2600	220	90	0,09	
87-94	30	7000	4500	5.6	7.1	2000	590	378	8.70	7.6	2700	300	91	0,11	
95-102	29	7000	4500	5.88	7.1	2000	490	370	9.28	7.6	2400	280	93	0,10	
103-115	26	7000	4500	6.48	7.1	2000	900	500	9.08	7.55	2400	400	87	0,15	

Table 2. Results obtained under different experimental conditions in the first-stage UAPBR

HRT (hour), COD (mg  $l^{-1}$ ); SS (mg  $l^{-1}$ ); OLR (g COD  $l^{-1}$  day<sup>-1</sup>); Alkalinity (mg CaCO<sub>3</sub>  $l^{-1}$ ); VFA (mg  $l^{-1}$ ); Gas Production l CH<sub>4</sub>  $l^{-1}$  day<sup>-1</sup>

Table 3. Results obtained under different experimental conditions in the second-stage UAPBR

Days	HRT	Influent parameters						Effluent parameters							
		COD	SS	OLR	рН	Alkalinity	COD	SS	Gas Production	pН	Alkalinity	VFA	COD Removal (%)	VFA/ Alkalinity	
0-18	74	2400	450	0.8	7.3	2000	1300	120	0.62	7.6	2100	130	46	0.06	
19-22	60	1350	410	0.54	7.2	2000	620	110	0.509	7.6	2200	140	54	0.06	
23-47	53	1600	400	0.73	7.3	1800	340	105	0.97	7.5	1900	135	78	0.07	
48-57	48	950	430	0.47	7.2	2050	150	105	0.671	7.6	2000	140	84	0.07	
58-65	53	900	410	0.49	7.3	2200	160	100	0.341	7.7	1950	140	82	0.07	
66-75	44	750	410	0.52	7.5	2250	145	100	0.706	7.7	2500	145	80	0.06	
76-86	36	700	395	0.54	7.5	2360	140	100	0.731	7.8	2400	160	81	0.06	
87-94	31	590	390	0.48	7.6	2600	125	105	0.641	7.7	2500	175	78	0.07	
95-102	30	490	380	0.42	7.6	2700	100	105	0.558	7.8	2650	210	79	0.08	
103-115	26	900	380	0.85	7.55	2650	260	125	0.521	7.5	2900	280	71	0.09	

HRT (hour), COD (mg l<sup>-1</sup>); SS (mg l<sup>-1</sup>); OLR (g COD l<sup>-1</sup> day<sup>-1</sup>); Alkalinity (mg CaCO<sub>3</sub> l<sup>-1</sup>); VFA (mg l<sup>-1</sup>); Gas Production l CH<sub>4</sub> l<sup>-1</sup> day<sup>-1</sup>

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