Bull. Environ. Contam. Toxicol. (1987) 38:9–14

Bulletin of Environmental Contamination and Toxicology

Aerobic Treatment of Wine-Distillery Wastewaters

D. Sales, M. J. Valcárcel, L. Pérez, and E. Martinez de la Ossa

Faculty of Sciences, University of Cádiz, Spain

Waste from food-processing and allied industries is largely made up of organic compounds which can be metabolised by aerobic or anaerobic means (Lora and Miró 1978). However, these wastes present a series of problems to biological depuration plants, such as the need for prior treatment to establish conditions suitable for the development of the microorganisms responsible for the process; and the long retention time of the biomass if acceptable effluents are to be obtained. Again, the seasonal nature of many of these industries makes for very heterogeneous waste. This means that treatment plant must be versatile and are subject to rapid successions of close-down and start-up interspersed with long intervals of inactivity. All these difficulties oblige the industries in this sector to adapt depurative technology to their particular needs.

Wine distilleries fall into this general category. Their waste (called vinasses) is acidic (Fiestas et al. 1981), has a high organic content (Valcárcel et al. 1982) and varies widely according to the raw matter distilled: wine, lies, etc. (Sales et al. 1982).

This paper studies the start-up of digestors for aerobic treatment of vinasses and the establishment of optimum operating conditions for an adequate depurative performance.

MATERIAL AND METHODS

The technique used was that of activated sludges. Completely mixed aerobic digestors without sludge recycle were used. Their capacity was 1.5 litres, while the volume utilised was 1.0 litres, to avoid the overflow of the foam produced in the process. The digestors were maintained at the optimum temperature of 25 $\pm$ 1°C (Fiestas 1983) by immersion in thermostatics bath. The medium was agitated by bubbling air into the digestors. Air flow (measured under normal conditions) was 5 litres per litre digestor per minute.

Send reprint request to Dr. E. Martinez de la Ossa at Facultad de Ciencias, Apdo. nº 40, Puerto Real (Cádiz), SPAIN.

Table 1. Mean physical-chemical characteristics of vinasses fed into digestors.

Parameters	Start-up of Digestors		Process Optimization	
	Unneutralized Vinasses	Neutralized Vinasses	Unneutralized Vinasses	Neutralized Vinasses
pH	3.20	7.53	3.29	7.61
COD (g O_2/l)	21.86	21.57	20.13	20.01
BOD (g O_2/l)	12.85	12.00	12.61	12.98
TS (g/l)	20.30	22.90	20.15	22.87
OS (g/l)	15.37	15.76	16.18	15.56
SS (g/l)	0.50	0.66	0.21	0.60
SOS (g/l)	0.44	0.55	0.15	0.45
TN (mg N/l)	306	308	335	335
TP (mg P/l)	56	49	59	58
MR (col.10^6/ml)	6.80	5.30	2.80	3.50

Untreated vinasses (acidic) and vinasses neutralized by adding NaOH 7N, were subject to aerobic treatment in parallel digestors. Table 1 shows the characteristics of the vinasses. All experiment were conducted in duplicate.

Each digestor, loaded with its particular type of vinasse, was started up by injection of an inoculum from a vinasses treatment plant using activated sludge. After this, the digestors received a daily infeed of 100 ml of vinasses (both neutralized and non-neutralized), while the same volume was taken off. The feed-rate was maintained for four weeks, the time necessary to attain constants percentages of organic matter degradation and microorganisms content, that is to say, to attain steady state conditions.

In order to achieve optimum depuration, a series of experiments were conducted at different retention times (retention time coincides with hydraulic retention time in this type of digestors). Thus it was possible to determine the minimum retention time needed for acceptable depurative performances, while providing stable conditions for the system to work in. The following method was followed in each of these experiments: first, the volume of feed was set in such a way as to obtain the requisite retention time, and was maintained for the 12 day's duration of each experiment; during a 5-days transition period, the characteristics of the effluents altered as consequence of the change in load density; after this period, the system stabilized and remained at steady-state conditions until the end of the experiment.

During these experiments, samples of effluents were collected and analysed, first just as they were, then after centrifuging at 1000 g for 5 minutes. The parameters determined in both influent and effluent are analysed according to the techniques described in Standard Methods (A.P.H.A. 1980). These parameters were: pH, COD, BOD, Total (TS), Suspended (SS) and Dissolved (DS) Solids, Total (TOS), Suspended (SOS) and Dissolved (DOS) Organic Solids, Total Nitrogen (TN), Total Phosphorous (TP), Dissolved Oxygen (DO) and Microbiological Recount (MR).

Table 2. Results of start-up of digestors fed with unneutralized vinasses (effluent not centrifuged).

Parameters	Time (days)							
	0	3	7	10	14	17	21	28
pH	3.20	5.95	5.60	5.53	5.02	7.40	7.49	8.15
COD (g O_2/l)	21.86	18.93	13.98	11.70	10.00	7.56	6.30	6.33
BOD (g O_2/l)	12.85	-	7.95	-	5.50	-	2.80	3.26
TS (g/l)	20.30	14.72	13.72	12.02	11.32	10.64	9.50	9.56
OS (g/l)	15.37	11.31	10.07	8.22	7.75	7.19	5.60	7.88
SS (g/l)	0.50	4.62	5.72	4.48	5.00	5.18	5.08	5.79
SOS (g/l)	0.46	4.51	5.36	4.28	4.40	4.48	4.43	5.42
TN (mg N/l)	308	302	302	291	291	291	305	328
DO (mg O_2/l)	4.50	0.50	0.40	0.60	1.50	1.20	1.30	2.35
MR (col.10^6/ml)	7.60	-	630	710	750	800	230	960

Table 3. Results of start-up of digestors fed with neutralized vinasses (effluent not centrifuged).

Parameters	Time (days)							
	0	3	7	10	14	17	21	28
pH	7.53	6.05	8.06	6.18	6.50	7.65	8.03	8.65
COD (g O_2/l)	21.57	16.31	13.97	10.54	9.88	6.30	6.30	6.03
BOD (g O_2/l)	12.00	-	7.36	-	6.10	-	2.80	3.21
TS (g/l)	22.90	17.24	16.35	13.68	14.31	15.34	12.17	13.75
OS (g/l)	15.76	11.23	10.79	8.55	8.46	8.99	6.07	8.50
SS (g/l)	0.66	4.32	5.68	5.24	5.34	5.65	5.31	5.67
SOS (g/l)	0.55	4.21	5.12	4.88	4.56	4.90	4.62	5.33
TN (mg N/l)	308	280	302	291	291	280	309	333
DO (mg O_2/l)	4.50	0.50	0.30	0.70	1.50	1.70	1.70	2.40
MR (col.10^6/ml)	5.60	-	800	700	940	910	220	150

RESULTS AND DISCUSSION

Tables 2 and 3 show the results obtained from analysis of the effluents during the start-up of the digestors. Table 2 is for the treatment of acidic vinasses, and Table 3 is for neutralized vinasses.

As can be seen from the tables, pH stabilized at around 8 in all the digestors, irrespective of the type of vinasses treated. In the case of acidic vinasses, this is due to two causes: on the one hand, the organic acids oxidize and are eliminated as CO_2; and the other hand, the salts oxidize to generate basic compounds. These, reacting with the CO_2 produced, form carbonates and bicarbonates, which generate a pH buffer in the medium, of between 8.0 and 8.3. In the case of neutralized vinasses, the second cause was operative in the rise in pH values.

The values of COD and BOD fell with time as a consequence of bacterial growth and the increase in the degrading capacity of the medium. The COD and BOD removals were stabilized at 70% and 75%, respectively, reaching 80% and 85% in centrifuged effluents.

There was a similar sequence for the total and organic solids contents of the effluents, where elimination stabilized at around 40-47%. These percentages reached 60% and 70% respectively where effluents were centrifuged. It is worth noting that there is a higher total solids content in the case of neutralized vinasses as a result of adding NaOH.

In all cases, suspended solids stabilized between 5 and 6 g/l after the first week. 90% of these solids are organic and make up the digestors biomass, an observation confirmed by the results of the microbiological recounts.

Total nitrogen content in all the digestors was similar to that of the original vinasses, which indicates that nitrogen is not eliminated from the medium. At the end of the start-up, 50-60% of total nitrogen was found to form part of the cellular constituents of the biomass. Also noted was a high nitrate presence (20-30 mg N/l) and some nitrites (less than 1 mg N/l) resulting from the oxidation of the ammonia generated in the deamination of nitrogenated components. This fact is confirmed by the low ammonia content (5-10 mg N/l) of the medium compared to that reached in anaerobic digestion (40-50 mg N/l) for similar vinasses (Valcárcel et al. 1984).

At the end of start-up, dissolved oxygen levels also stabilized owing to the sustained cell-growth rate in the medium.

Tables 4 and 5 show the results of the analysis of the effluents from the digestors in the series of the experiments designed to determine the retention times which will give optimum operating conditions.

In these tables it can be seen that in all the digestors, pH values fell as the retention time decreases. Where acidic vinasses are used, this fall was sharper because of the acidity of the vinasses.

COD and BOD values reached minimum up to 8 days of retention time, and maintained that level over longer periods. This was due to the presence of compounds like polyphenols, which are difficult for the aerobic flora to break down (Bories 1982). For retention times of between 8 and 20 days, the COD and BOD removals were of 77% and 88%, respectively. These values are comparable to those obtained by other authors in aereated lagoons (Micheli 1982) and with activated sludges (Bories et al. 1981), in both cases with retention times of 15 to 20 days.

There were similar developments with the effluents' solids content (total, organic, total dissolved and dissolved organic) since these are subject to the same effects. Solids contents were higher for the treatment of neutralized vinasses because the addition of NaOH. The level of dissolved oxygen in the medium fell as the retention time was decreased, or put in another way, as the load

Table 4. Results obtained on optimizing operating conditions of digestors with unneutralized vinasses feed.

Parameters		Retention Time (days) 20	10	8	6	5	4	3	2
		Uncentrifuged effluents							
pH		8.41	8.15	6.93	6.61	5.53	5.14	4.96	4.43
COD	(g O_2/l)	5.88	6.66	7.35	8.87	10.14	12.42	14.27	16.44
BOD	(g O_2/l)	2.91	3.26	4.10	5.51	6.43	7.20	8.08	9.18
TS	(g/l)	10.90	10.56	11.31	12.40	14.06	15.10	15.14	15.93
OS	(g/l)	7.98	7.88	8.67	9.52	10.95	11.30	10.80	11.46
SS	(g/l)	6.09	5.79	6.18	6.15	6.57	5.80	4.00	3.43
SOS	(g/l)	5.68	5.42	5.76	5.56	6.21	5.17	3.52	2.60
TN	(mg N/l)	316	328	327	310	298	291	290	295
DO	(mg O_2/l)	2.45	2.35	1.90	1.30	0.50	0.50	0.50	0.60
MR	(col.10^6/ml)	1600	960	1300	910	890	820	640	360
		Centrifuged effluents							
COD	(g O_2/l)	2.97	3.33	4.03	5.06	5.61	7.50	9.47	13.64
BOD	(g O_2/l)	1.31	1.55	1.65	2.74	3.29	4.38	4.99	7.98
DS	(g/l)	4.81	4.77	5.13	6.25	7.49	9.30	11.14	12.50
DOS	(g/l)	2.30	2.46	2.91	3.96	4.74	6.13	7.28	8.86
TN	(mg N/l)	145	132	157	156	180	189	203	221

Table 5. Results obtained on optimizing operating conditions of digestors with neutralized vinasses feed.

Parameters		Retention Time (days) 20	10	8	6	5	4	3	2
		Uncentrifuged effluents							
pH		8.50	8.65	8.12	7.56	7.78	7.50	7.36	7.15
COD	(g O_2/l)	5.91	6.03	6.42	8.17	9.97	11.84	14.05	15.60
BOD	(g O_2/l)	2.98	3.21	3.77	5.35	6.06	6.64	8.02	9.05
TS	(g/l)	14.02	13.75	13.33	13.71	16.63	17.09	18.01	18.02
OS	(g/l)	8.64	8.56	8.47	8.76	10.58	11.38	11.80	12.49
SS	(g/l)	6.02	5.67	5.13	5.45	6.76	6.15	5.75	4.40
SOS	(g/l)	5.57	5.33	4.66	4.78	6.83	5.86	5.08	4.03
TN	(mg N/l)	315	333	320	312	309	302	297	289
DO	(mg O_2/l)	2.60	2.40	1.30	1.00	0.60	0.70	0.40	0.50
MR	(col.10^6/ml)	1900	1500	1900	2000	940	800	730	370
		Centrifuged effluents							
COD	(g O_2/l)	3.40	3.59	3.98	4.65	5.49	7.14	8.93	11.52
BOD	(g O_2/l)	1.38	1.57	1.83	2.59	3.24	4.19	5.03	6.98
DS	(g/l)	8.00	8.08	8.20	8.26	9.87	10.94	12.26	14.62
DOS	(g/l)	3.07	3.23	3.81	3.98	4.75	5.52	6.72	8.46
TN	(mg N/l)	141	153	146	156	172	186	206	224

density fed to digestors (g COD/l/day) increased, because of the higher oxygen demand of the microorganisms in breaking down the organic matter. However, the fall in oxygen levels brings with it a reduction in the number of the microorganisms in the medium, a fact confirmed by the microbiological recount and the value of

suspended organic solids. If load density increases (retention time decrease) a point is reached where the rates of regeneration of the flora and of evacuation of microorganisms in the digestors are equal. Here, the effect known as "wash-out" ensues, and consequently the nullification of the depurative capacity of the digestors.

Once the digestors reach steady-state, 70-75% removals of COD and BOD are attained. These values rise to 80-85% when the effluents are centrifuged. Moreover, neutralization of vinasses does not improve depurative performance, which simplifies the process and reduces operating cost.

Optimum retention time for aerobic treatment of vinasses is 8 days. With this retention time, the effluents show pH-values between 6.5 and 8.0, COD and BOD removals of 78-88%, dissolved oxygen contents of over 1 mg/l, and microorganism populations of over 10^9 col/ml.

REFERENCES.

A.P.H.A., A.W.W.A., W.P.C.F. eds. (1980) Standard methods for the examination of water and wastewater, 15th ed., Washington.

Bories A (1982) Méthanisation des eaux résiduaires de distilleries vinicoles. Ind Alim Agric 99(4): 215-225.

Bories A, Raynal J, Manzenet J (1981) Performances et coûts de la depuration biologique aerobie des eaux residuaires de distilleries vinicoles. Resultats industriels sur vinasses de vin blanc. Techn Sci Munic Eau 11: 539-546.

Fiestas JA (1983) Curso de calidad de aguas. Servicio de publicaciones de la Universidad de Cádiz. Cádiz, Spain.

Fiestas JA, León R, García A, Fernández JR, Sainz JA (1981) Aplicación de los procesos anaerobios en la depuración de las aguas residuales industriales de alta carga orgánica. Ing Quim 147(6): 85-89.

Lora F, Miró J (1978) Técnicas de defensa del medio ambiente, ed. Labor, Barcelona, Spain.

Micheli A (1979) Depurazione delle acque reflue da distilleria mediante digestione anaerobia e succesiva ossidazione biologica. R.P.A. Risorce Ambientali, Perugia, Italy.

Sales D, Valcárcel MJ, Perez L, Martinez de la Ossa E (1982) Determinación de la carga contaminante y naturaleza de los vertidos de destilerías de alcohol de vino y alcohol vínico. Quim e Ind 28(10): 701-706.

Valcárcel MJ, Pérez L, Sales D (1984) Obtaining suitable methanogenic flora for anaerobic treatment of vinasses from distilleries. 3rd Mediterranean Congr Chem Eng, Barcelona, Spain.

Valcárcel MJ, Sales D, Pérez L, Martínez de la Ossa E (1982) Estudio de las aguas residuales de destilerías de alcohol de vino y alcohol vínico. Proc 2nd Jor Univ Jerez, 474-492, Cádiz, Spain.

Received April 10, 1986; accepted September 2, 1986.