

# ANAEROBIC TREATMENT OF PECTIN WASTES: EXPERIENCES WITH A FULL SCALE AND A SEMI TECHNICAL PLANT

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## ABSTRACT

Pectin wastes are treated in a full scale anaerobic treatment plant which was built after extensive pilot test studies had been performed. The process used is an anaerobic activated sludge system. Between the reactor and the settling tank the mixed liquor passes through a vacuum degasser. This results in a proper settling of the return sludge. The efficiency of treatment is shown by a COD removal of 88 % and a BOD<sub>5</sub> removal of 90 %.

## KEYWORDS

Industrial wastes; anaerobic treatment; anaerobic activated sludge system; vacuum degasser.

## 1) Introduction

In autumn of 1982 a plant for the treatment of industrial effluent from pectin production was put into operation. It involved an anaerobic activated sludge plant conceived upon the basis of data compiled through extensive preliminary testing. The tests were conducted from 1976 to 1978 whereby two separate test sites were involved in succession, each on a semi technical scale. The first site possessed a digester volume of 3 m<sup>3</sup>, the second site 4,5 m<sup>3</sup>. The tests confirmed the fundamental viability of the anaerobic process for the purification of pectin wastewater and furthermore yielded data concerning necessary detention time and achievable effluent specifications.

## 2) Description of the industrial effluent and treatment plant

The industrial effluent is composed of approx. 70 % effluent from distillation processes, approx. 25 % rinsing water from the production hall, and approx. 5 % water from boiler blow downs. The treatment plant is designed for a maximal hourly flow of  $33 \text{ m}^3/\text{h} \cong 792 \text{ m}^3/\text{d}$ . The characteristic parameters (24 h composite samples) of the total effluent are given in Table 1.

parameter	minimal	50 % of values less than	80 % of values less than	maximal
BOD <sub>5</sub> (mg/l)	2.400	4.250	5.800	10.100
COD (mg/l)	4.000	9.600	13.800	27.500
KMnO <sub>4</sub> (mg/l)	9.800	20.100	24.700	31.100
Total N (mg N/l)	400			1.600
Phosphate (mg PO <sub>4</sub> /l)	2	13	17	22
pH	1	2	2	11

Table 1: Characteristics of the industrial effluent,  
24 h composite samples

The various component currents from the factory are fed into a neutralisation station (volume  $V = 25 \text{ m}^3$ ). The hot, neutralized effluent is passed through a heat exchanger in which it is cooled to a temperature necessary to achieve mesophilic range values of  $36^\circ \text{C} - 39^\circ \text{C}$  in the anaerobic reactor. The effluent then flows into an  $80 \text{ m}^3$  mixing tank where a preacidification takes place. A denitrification in the mixing tank through use of aerobic excess sludge is to be included at a later time.

The further flow sheet of the effluent corresponds to the diagram in Fig. 1. The effluent is injected by two non clogging centrifugal pumps into a circulation cycle driven by the same pump type. This segment of the circulation pipe line is constructed as a mixing jet in front of which the discharge points of the return sludge and a steam line are located. The steam can, if necessary, be employed to heat the reactor contents.

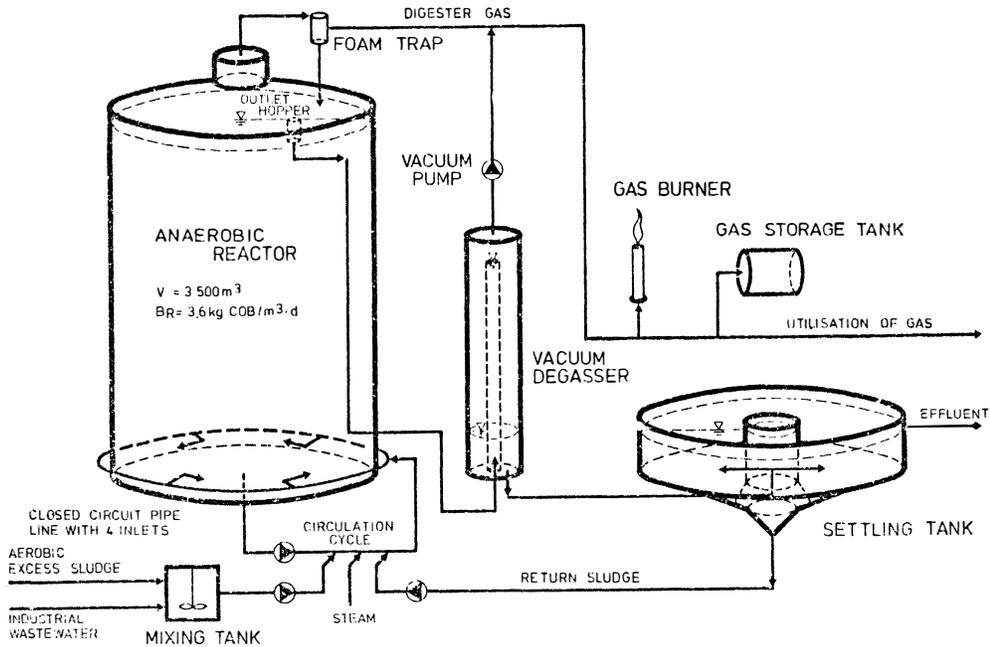


Fig. 1: Flow sheet of the anaerobic treatment plant

The pressure side of the circulation cycle empties into a closed circuit pipe line on which four large inlets into the reactor are equidistantly located, the orifice of each inlet directed towards the next. The circulation current can be injected into the reactor through all four inlets simultaneously or through any one of the four inlets, in which case a switching program permits regulation of injection sequence and duration. The program signals electric gate valves which modulate individual inlet status. The suction side of the circulation cycle can optionally be fed through an opening in the reactor base or through an intake line which reaches into the upper third of the reactor.

The anaerobic reactor is a round welded steel container 23,50 m in height and 14.00 m in diameter, the interior surface of which is covered with a tar epoxy coating for corrosion protection.

The outflowing anaerobic mixed liquor is passed through a gravity tube into a vacuum degasser consisting of a 12 m vertical twin tube whereby the reactor discharge runs down the outside of the inner tube through which the discharge has entered the degasser. Liquid seal gas pumps create a partial vacuum in the degasser of 70 - 100 mbar.

The degassing provides for the trouble free sedimentation of anaerobic sludge, otherwise impaired by residual or newly formed gas, in a 480 m<sup>3</sup> round settling tank with a surface area of 175 m<sup>2</sup> and an average depth of 2,75 m. Non clogging centrifugal pumps transport return sludge from this point through the mixing jet mentioned above into the circulation line. Excess sludge is removed directly from the return sludge flow by means of eccentric screw pumps.

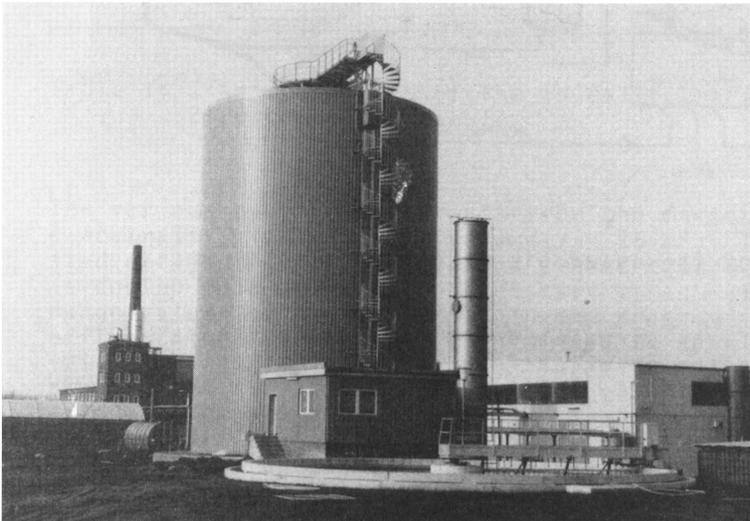


Fig. 2: Total view of the anaerobic plant

Digester gas from the reactor and vacuum degasser is fed into a gas compressing station and from there into the boilerhouse. Short term fluctuations in production and consumption can be compensated by the gas storage tank ( $V \approx 65 \text{ m}^3$ ); venting is achieved through the waste gas burner.

Fig. 2 shows the whole treatment plant with the settling tank in the foreground. On the right next to the reactor is the vacuum degasser and on the left one can see the gas storage tank. All liquid and gas lines as well as fittings are of stainless steel.

### 3) Filling and start up

In the latter part of October 1982 the reactor was brought up to a fill level of approx. 1.900 m<sup>3</sup> with drinking water at 39,5° C. On Nov. 1 a supplement of 20 m<sup>3</sup> digester sludge from a nearby municipal treatment plant was applied in order to achieve a rapid reduction in O<sub>2</sub> content within the reactor.

The following day the reactor was loaded for the first time with 30 m<sup>3</sup> industrial effluent and by midday the oxygen content in the tank came to 4.5 mg O<sub>2</sub>/l. In the course of Nov. 3 the O<sub>2</sub> content fell to 0,2 mg O<sub>2</sub>/l so that from Nov. 4 on the reactor was able to be seeded with 100 m<sup>3</sup> digester sludge daily for 5 days. The sludge was delivered in tank trucks and freed of coarse matter by means of a perforated trommel revolving screen before application to the reactor.

The daily industrial effluent flow as well as reactor temperature, pH, and organic acid content are listed in Table 2. The COD concentration of the effluent was relatively constant at 16.000 mg/l.

Two weeks after the initial influx of digester sludge, the reactor attained full volume and from that point on an influent equivalent volume of warm water was displaced into the originally half full settling tank. The approach of initially filling the reactor with only 1.900 m<sup>3</sup> was taken in order to minimize premature losses of biomass from the system and to counteract cooling of reactor contents by return sludge as influenced by winter temperatures.

During the start up, which lasted until about mid February, the level of incoming industrial effluent was carefully coordinated with parameters critical for biological processes within the reactor, i.e.:

- a) organic acid content;
- b) pH;
- c) CO<sub>2</sub> component of digester gas.

Whereas the organic acid content continuously rose, reaching a peak of 2.500 mg (as acetic acid)/l on Jan. 21, 1983, the other two parameters displayed no discernable trend. The pH varied between 6.75 and 7,40, strongly fluctuating with-

date	approx. daily amount of industrial effluent	reactor temperature	reactor organic acids (acetic acid)	pH in reactor	daily addition of digester sludge	remarks
(1982)	(m <sup>3</sup> /d)	(°C)	(mg/l)	(-)	(m <sup>3</sup> /d)	(-)
01.11.	-	39,75			20	reactor filled with 1.900 m <sup>3</sup> drinking water
02.11.	30	39,75			-	first influx of effluent
03.11.	70	39,75	49		-	oxygen content 0,2 mg O <sub>2</sub> /l
04.11.	25	39,50	60	6,99	100	
05.11.	45	38,60	84	7,02	100	
06.11.	55	37,70	96	7,07	100	
07.11.	60	38,10	126	7,12	100	
08.11.	75	37,30	135	7,16	100	addition of digester sludge terminated
09.11.	85	36,50	155	7,20	-	
10.11.	90	36,30	160	7,21	-	
11.11.	100	36,00	160	7,20	-	
12.11.	110	36,00	160	7,08	-	
13.11.	120	37,40	160	7,15	-	
14.11.	130	38,00	160	7,15	-	reactor reached full volume

Table 2: Amounts supplied, reactor temperature, organic acid concentration, and pH in the first two weeks after start up of the anaerobic reactor.

in a few days; the  $\text{CO}_2$  component of the digester gas varied between 27,5 and 51,0 %. The time of gas production onset could not be established because up until the filling date the system was open over the emergency overflow. The gas storage tank began to fill immediately with the switch to closed system conditions on Nov. 14, and on the evening of Nov. 15 the waste gas burner was successfully ignited for the first time.

The organic acid concentration fell from 2.500 mg/l to around 250 mg/l over the course of the two weeks starting Jan. 21 so that the effluent amount could be increased. The plant has been operating daily since mid February with loads between 400 and 550  $\text{m}^3/\text{d}$ .

#### 4) Operating results to date and comparisons with results from semi technical scale experiments

The average daily flow for the sample months evaluated here of March, April and May was 480  $\text{m}^3/\text{d}$ , which corresponds to a detention time of 7,3 d. The COD influent concentration (mixed) averaged 12.000 mg/l which corresponds to a COD space loading of  $B_R = 1.65 \frac{\text{kg COD}}{\text{m}^3 \cdot \text{d}}$ . An average operating

efficiency of  $\eta_{\text{COD}} = 88 \%$  was achieved for the reactor, corresponding to an average effluent concentration (mixed) of 1.400 mg/l. The maximum effluent COD concentration was approx. 2.300 mg/l, the minimum slightly under 1.000 mg/l. (In pilot tests conducted in 1978 with the 4,5  $\text{m}^3$  anaerobic reactor, the effluent COD concentration averaged 2.000 mg/l at a detention time of 6 days.) The  $\text{BOD}_5$  space loading averaged  $B_R = 0,75 \frac{\text{kg BOD}_5}{\text{m}^3 \cdot \text{d}}$ .  $\text{BOD}_5$  contents were reduced from an average of 5.400 mg/l (mixed) to values between 350 and 1.200 mg/l (mixed) or an average of 500 mg/l (values averaged for May and June as only here were extensive  $\text{BOD}_5$  measurements undertaken). This corresponds to an efficiency of  $\eta_{\text{BOD}} = 90 \%$ .

The organic acid content varied between 100 and 500 mg/l. The reactor effluent displayed a pH 0,1 - 0,25 higher than that measured in the circulation line. The circulation line pH averaged over the 3 month period in question 7.2 and fell only on 5 days under 7.0 to 6.9. In the 80  $\text{m}^3$  mixing tank upline, as mentioned, from the reactor, a preacidification takes place which, together with the conversion into organic acids in the reactor (particularly at the start) and due to the upflowing current mentioned below, exerts a disproportionately greater influence upon the pH in the circulation line than upon that of the effluent.

Since the reactor is fed from the bottom out of the closed circuit pipe line and its outlet is located at the top, a deliberate upflowing current is generated in which lighter suspended solids are carried upwards whereas heavier ones remain in the lower position (whereby any possible sedimentation on the reactor floor is guarded against by the sub-horizontal inclination of the inlets). This stratification of suspended matter is evidenced by concentrations of suspended solids in the circulation line 25 - 50 % higher than those in the reactor effluent. On the whole, the total suspended solids in the circulation line increased during the 3 months indicated from an initial 20 g/l to 55 g/l, as until May 10<sup>3</sup> no excess sludge was removed. Subsequently, approx. 30 m<sup>3</sup> excess sludge was removed each weekday which brought the total suspended solids (in the circulation line) down to approx. 50 g/l. The volatile suspended solids dropped from 40 % (early March) to 25 % (end of May). This marks an enrichment of the sludge with inorganic material, primarily lime precipitation stemming from the neutralisation. In order to effect a decrease in this component and to hold it at lowest possible future levels, the neutralisation has been carried out since mid May exclusively with sodium hydroxide instead of lime.

Mixing in the reactor from the circulating pumps and from gas production was studied by means of tracer tests. It was determined that inflowing effluent is uniformly distributed over the entire reactor contents within 4 hours. This represents only approx. 2 % of the theoretical detention time, thus characterizing the effectiveness of the mixing process.

Due to an inorganic component of 60 - 75 %, the sludge volume index amounted to between 20 and 50 ml/l so that there were no settling problems at all.

A one time study of the settling tank yielded the suspended solids data in Fig. 3. The concentration of suspended solids in the upper 2 m of the supernatant shows no discernable increase with depth. The upper edge of the sludge lies 2 to 3 m deep; the sludge concentration at 3 m is 20 g/l and in the hopper bottom 44,8 g/l. The effluent dry solids content in this measurement was 150 mg/l, though normally averaging 110 mg/l with an average settleable solids concentration of 1.0 ml/l. The washed out suspended solids had a volatile component of 75 %, higher than that of the activated sludge in the reactor.

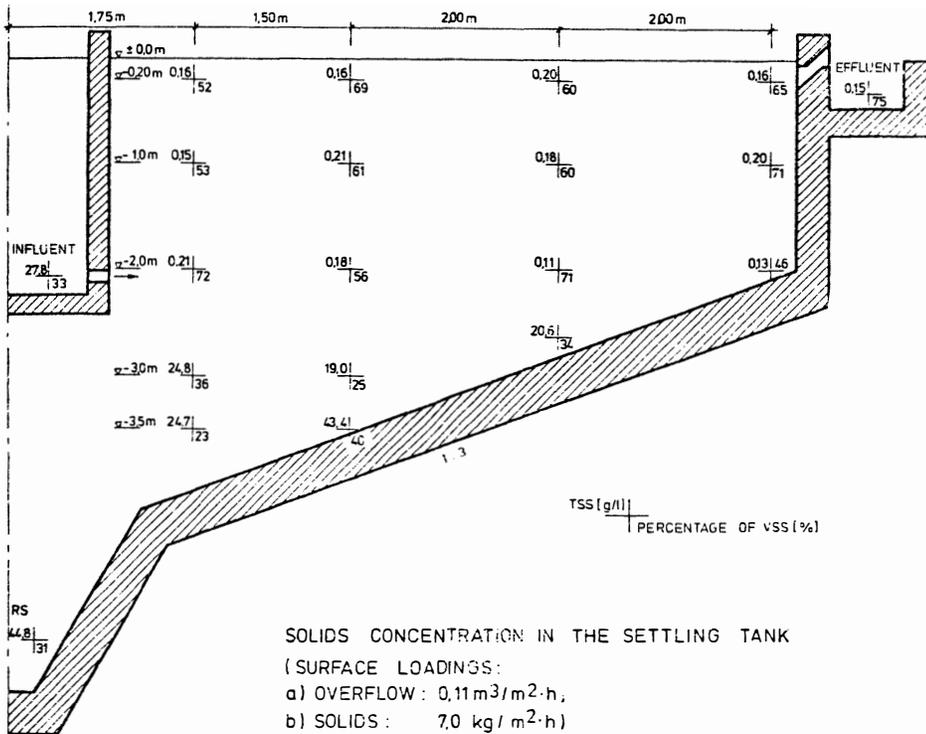


Fig. 3: Suspended solids concentrations at various depths in the settling tank.

Hinging upon an accurate orifice calibration, the daily gas production came to 2.600 m<sup>3</sup>/d resulting in a very high specific gas production of about 500 l/kg COD removed; CO<sub>2</sub> comprised an average 36.5 % of gas produced. Further study in the near future will reveal if gases other than CO<sub>2</sub> and methane, such as nitrogen for example, are present in significant quantities.

Nitrogen is present in the effluent at approx. 930 mg N/l, primarily as nitrate (calculated from the nitric acid consumption in the production process).

A total of 23 24-hour composite samples from the settling tank effluent were analyzed for nitrogen content. The following arithmetic means were obtained with only slight variations in the analyses.

organic N (mg/l)	NH <sub>4</sub> -N (mg/l)	NO <sub>3</sub> -N (mg/l)	total N (not incl. NO <sub>2</sub> -N) (mg/l)
69,4	96,5	1,1	167

Table 3: Nitrogen concentrations in the settling tank effluent (average of 23 values)

These results are surprising in that ammonification was anticipated in view of the on site results obtained 1978 from the one stage semi technical scale installation (4,5 m<sup>3</sup>) listed in Table 4.

	org. N (mg/l)	NH <sub>4</sub> -N (mg/l)	NO <sub>3</sub> -N (mg/l)	NO <sub>2</sub> -N (mg/l)	total N (mg/l)
influent	192	65	326	4	587
effluent	73	505	6	0	584

Table 4: Nitrogen concentrations in semi technical scale tests (4,5 m<sup>3</sup> digester)

A comparison of effluent values from Tables 3 and 4 shows similar concentrations of organic and nitrate nitrogen, whereas total nitrogen in the present full scale plant is much lower, a consequence of the lower ammonium-N concentrations.

Thus, a denitrification takes place in the reactor, whereby approx. 760 mg N/l is obviously converted to elemental nitrogen. 3 gas analyses, in which methane, CO<sub>2</sub>, and H<sub>2</sub> components were quantitatively measured, revealed a deficit of an unidentifiable gas, the amount of which would coincide with the amount of gaseous nitrogen produced by a denitrification of 760 mg N/l.

Since start up a heat production has been observed in the reactor. It became evident as the temperature in the reactor rose even though the temperature of the influent was lower than that in the reactor (despite the cooling effect of the return sludge). Table 5 shows the result of a thermal balance calculation for the months of Jan. to June 1983, whereby thermal losses due to radiation as well as to the addition of thermal energy due to the various pumps were taken into account. The basis for the calculation was provided by the temperature records for the influent and return sludge in addition to the two thermometers installed within the reactor. The production of heat can be expressed relative to the reactor contents (lines 1 - 4) or the influent (line 5). The measured heat production in line 1 is indicated as such whereas in lines 4 and 5 the data from lines 2 and 3 was brought into the calculation. According to line 5 a very substantial 23.6 to 43.9 kJ thermal energy/l influent was produced. According to MUDRACK (1), this is tentatively explicable through the microbial activity of facultative organisms which respire nitrate-bound oxygen. Table 6 shows the amount of heat expected for the assumption of denitrification and for the assumption of ammonification, both based on a daily flow of 500 m<sup>3</sup>/d.

No.		Jan.	Febr.	March	April	May	June
1	measured heat production (°C/d) 1), 3)	0,504	0,658	1,109	0,939	1,138	1,459
2	heat lost through radiation (°C/d) 1)	0,190	0,210	0,19	0,15	0,13	0,13
3	heat furnished through pumps (°C/d) 1)	0,160	0,160	0,17	0,18	0,19	0,20
4	calculated heat production (°C/d) 1), 4)	0,534	0,708	1,089	0,909	1,198	1,389
5	calculated specific heat production (°C/m <sup>3</sup> ) 2), 4)	5,65	6,29	7,80	6,90	8,78	10,50

1) relative to reactor contents

3) measured

2) relative to reactor influent

4) with allowance for lines 2 and 3

Table 5: Heat production in the anaerobic reactor from January to June 1983.

No.		Jan.	Febr.	March	April	May	June
1	calculated $\text{HNO}_3$ - concentration in effluent (mg/l)	3684	3684	4392	4470	4049	4533
2	heat production 1) through denitrification ( $^{\circ}\text{C}/\text{m}^3$ )	4,8	4,8	5,7	5,8	5,3	5,9
3	heat production 1) through ammonification ( $^{\circ}\text{C}/\text{m}^3$ )	7,7	7,7	9,2	9,3	8,5	9,5

1) relative to influent

Table 6: Theoretical heat production through denitrification or ammonification from January to June 1983.

By comparing the heat produced through denitrification (Table 6, line 2) with actually measured heat production (Table 5, line 5), a deficit results which would have to be accounted for by other factors.

Basically, it should be noted that in the operating results to date the high COD efficiency of  $\eta = 88\%$  and the high  $\text{BOD}_5$  efficiency of even  $\eta = 90\%$  represent quite positive aspects. Future operating results will have to demonstrate whether or not and to what extent these efficiencies will decline with increased space loading accompanied by shorter detention times.

#### 5) Plant construction costs

The total costs for the plant (incl. such components as neutralisation tank, denitrification tank, and gas storage tank) come to 2,8 million DM. Of that, DM 472.000 go towards the steel carcass work, DM 110.000 towards thermal insulation work, and DM 320.000 towards the complete settling tank including scraper.

If the total costs are taken in relation to the container volume, the figure of 800 DM/m<sup>3</sup> is obtained. The specific cost of the settling tank is 665 DM/m<sup>3</sup>.

On the basis of 330 operating days per year, an assumed depreciation period of 10 years, and an interest rate of 9 %, the following specific investments are arrived at for the project:

- COD : 0,13 DM/kg COD removed;
- BOD<sub>5</sub> : 0,31 DM/kg BOD<sub>5</sub> removed.

## Literature

- (1) Mudrack, K.                      Erwärmung des anaeroben Reaktors durch biologische Ammonifikation oder Denitrifikation des Nitrats.  
August, 1983                      unpublished

## DISCUSSION

Mr. E. I. Clark (North West Water Authority U.K.) asked what the mixing power input was during start-up and whether phosphorus was retained in the recycled sludge to offset the very high C/P ratio in the feed liquor. When using caustic soda in place of lime for pH control had any significant thermal gradient effects been observed which had lead to excessive loss of solids with the final effluent despite vacuum degassing? Could the Authors indicate the present ratio of input COD to volatile solids produced in the reactor and the subsequent treatment given to the final effluent before discharge to a watercourse.

Mr M Spiegel, (Consultant, USA) referred to the Anaerobic Contactor, also sometimes called an Anaerobic Filter. The present development of the Anaerobic Contactor (Filter) used contact of comparatively high strength wastewaters, such as supernatants from thermal treatment and dewatering of sewage sludge and biological reduction of industrial wastes. Demonstrated feeds are 1000 to 50,000 mg/l COD, resulting in 65 - 85% reduction at loadings of greater than 1,000 lb. COD/1000 Cf/day. (15 kg/m<sup>3</sup>/day.)

The general form of the Anaerobic Contactor is a cylindrical reactor packed with plastic ball rings of about 1.2 cm diameter on which a biological slime was produced.

The comparative advantages of the Anaerobic Contactor to aerobic processes were: 1 - energy credit - gas generation. 2 - High treatment efficiency - 3 - Low operation cost. 4 - Low capital cost. 5 - Minimal operation attention. 6 - Negligible sludge generated. 7 - Shock load absorptive.

The Anaerobic Contactor made heat treating competitively economical by low cost, effective treatment of supernatant liquor and could serve as a failsafe emergency treatment system to handle sludge liquors, etc. where bypassing was prohibited. Most usually process difficulties were associated with high strength liquors.

An example of a highly effective and economical application of an Anaerobic Contactor was for treatment of the distillery wastes from the Bacardi Rum Catano Plant, the world's largest rum distillery, near San Juan, Puerto Rico. The Waste processing was based on a self sustaining reaction producing methane and an inert effluent which was environmentally assimilated by the nearby Atlantic Ocean.

The rum distillery wastes of 0.25 MGD ( $950 \text{ m}^3/\text{day}$ ) contained 36,000 - 42,000 mg/l BOD; 85 - 105,000 mg/l COD; 4 - 8,000 mg/l TSS; pH<sub>6</sub> of 4.5 - 5.0. The Anaerobic Filter (Contactor) comprised a  $13,250 \text{ m}^3$  Anaerobic Filter tank packed with Goodrich Vinyl Core Meda, holding a fixed film of about 3mm. The contact process started up quickly as well as idled. Loading was approximately 175 lb. DOD / 1,000 CF/Day of tank capacity. ( $2.8 \text{ KG}/\text{m}^3/\text{day}$ ).