#### V EXPERIMENTAL RESULTS AND DISCUSSION

### 5.1 Characteristics of Tapioca Starch Wastes

Table 10 summarizes essential characteristics of the waste samples used in the experimental study. From the results the following conclusions may be drawn:

- a) Temperature of the wastewaters was in a range between 26° and 30°C which was a normal range of ambient temperature experienced in Thailand. Hence, anaerobic treatment of tapioca starch wastes can be carried out in the mesophilic range with no heating requirement.
- b) The wastewaters were acidic as indicated by their low pH values ranging from 3.9 to 5.2 and by zero alkalinity. It would be expensive to control the pH in treating tapioca starch wastes.
- c) The wastewaters were very turbid containing about 30-100 mg/l of settleable solids and about 1,700-3,920 mg/l of suspended solids.

  The total solids content varied from 3,560 to 6,800 mg/l. Apparently, about half of the solids were in soluble and colloidal forms.
- d) The wastewaters were high in organic content. The first-separator waste was more concentrated than the combined wastes as would be expected. The COD and BOD<sub>5</sub> values varied respectively in a range from 5,100 to 7,760 mg/l and 3,120 to 6,650 mg/l. The COD:BOD ratio was on the average bout 1.3. It can be inferred that tapioca starch wastes can be easily treated using a biological process and that the BOD<sub>L</sub> can be approximated by the COD.
- e) The COD:N:P ratio was on the average about 100:2:0.1.
  When this ratio was compared with the ratio of 100:1.1:0.2 for anaerobic treatment as recommended by Mc CARTY (1964), it was apparent that the wastewaters were slightly deficient in phosphorus. Consequently, phosphorus supplement may be necessary in the treatment.

Table 10 Characteristics of Tapioca Starch Wastes

	Song Charc	en,Chonburi	Song Charoen	Tai Wah	Sahaperch	Range of
Analysis	First Separator	Combined Wastes	Rayong *	Rayong *	phol Tawanok Chonburi*	Combined Wastes
Temperature 'C	28-30	26-30	28	26	28	26-30
pH	4.1-4.5	3.9-4.8	4.0-5.2	4.1	4.3	3.9-5.2
Alkalinity (mg/l as CaCO3)	0	0	0	0	0	0
Suspended Solids (mg/l)	3,400-4,200	2,170-3,920	1,700-3,780	2,170	3,020	1,700-3,92
Total Solids (mg/l)	5,310-6,400	3,840-5,100	3,560-6,800	4,930	5,610	3,840-6,80
Settleable Solids (mg/1)	40-110	30-65	45-100	55	80	30-100
BOD <sub>5</sub> (mg/1)	6,700-9,950	4,000-6,200	3,120-5,300	6,650	-	4,000-6,65
COD (mg/l)	8,050-13,200	5,100-6,880	4,700-6,900	7,760	6,200	5,100-7,76
Volatile Acids (mg/l as HAc)	450-850	570-1,200	450-1,200	-	-	450-1,20
Ammonia - N (mg/1)	4-12	2-20	3-22	8	11	2-22
Organic - N (mg/1)	180-340	100-220	95-150	130	145	95-220
Phosphorus (mg/1)	3-16	3-12	3-14	5	4	3-14

<sup>\*</sup> Combined Wastes

# 5.2 Filter Start-Up

In starting up the filter was heavily seeded with anaerobic-digested activated sludge as recommended by YOUNG and Mc CARTY (1969). The sludge was collected from a waste treatment plant operated by Thai Pure Drinks Ltd. in Bangkok. About 15 liters of the sludge was injected into the lower part of the filter column then the column was filled with the digester supernatant.

The filter was then operated continuously for 50 days to build up and acclimatize the bacterial flocs. The 50 day start-up period was divided into three phases. During the first 20 days the filter was fed with the digester supernatant having COD values from 600 to 1,000 mg/l. In the second 15 day phase the feed was changed to a 1,000 mg/l COD synthetic waste prepared from tapioca starch. In the last 15 days the filter received actual tapioca starch wastes diluted to reduce the COD to 1,000 mg/l.

Through out the start-up period the filter was operated under favourable environmental conditions. The feed rate was kept constant at 2 liters/hour and the COD:N:P ratio was controlled at a minimum value of 100:1.1:0.2 at all times. The pH was controlled in a range of 6.7-7.2 by adding 2,000 mg/l of sodium bicarbonate into the influent to act as buffer. Alkalinity, volatile acids, and COD of the influent and the effluent were analysed twice a week. Volume of the gas produced and percent methane content, were daily recorded in the last period. Results of the experiment were plotted in Figure 15.

Figure 15 clearly shows that immediately after the beginning of feeding the filter with the digester supernatant the COD removal steadily increased reaching 88% on the 20th day. When the supernatant was replaced with the synthetic waste the efficiency slightly dropped off to 84% on the 26th day but increased to 89% on the 35th day. When the filler received diluted tapioca starch wastes the COD removal

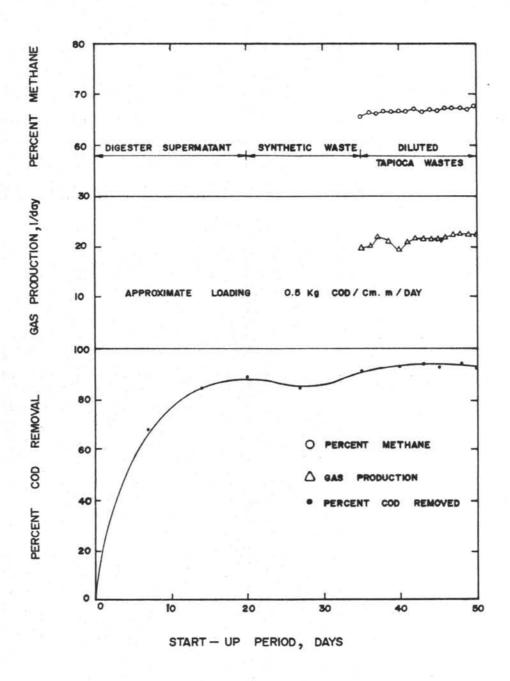


FIGURE 15 . PERCENT COD REMOVAL, GAS PRODUCTION AND METHANE

CONTENT DURING START - UP PERIOD

increased to 92% and remained constant through out the last 15 days. This indicated that the anaerobic filter was already under steady-state conditions and the microorganisms were well acclimatized to the tapiocastarch wastes.

Measurement of the production rate of the gas and its methane content was not made until after 30 days of operation due to the unavai-lability of apparatus. The rate of gas production was found to be relatively constant during the period of constant COD removal. This supported, according to YOUNG and Mc CARTY (1969) the previous conclusion that the filter was under steady-state conditions.

## 5.3 Filter Performance Under Controlled Conditions

After the start-up period the filter was operated under controlled environmental conditions for 32 days. The experimental runs were conducted as detailed in Table 11. The pH was controlled in a range between 6.6 to 7.2 using sodium bicarbonate buffer. Nutrient supplement was practised only during the first 7 days since after that nitrogen and phosphorus were always present in the effluent.

Each experimental run ranged from 7 to 10 days of which the first few days were required by the filter to attain steady-state conditions. For the first experimental run the organic loading applied was 0.6 kg COD/-cu.m./day. For subsequent experimental runs the applied loading was about twice of the preceded loading. Hence, response of the filter to shock loads could also be studied. The loading was varied by changing the COD while the feed rate and the detention time were kept constant at 42 liters/hour and 24 hours respectively.

Table 11 Effluent Quality and Treatment Efficiency During Steady-state Treatment of Tapioca Starch Waste

Influent St	Time of Steady-state	Theoretical Detention	Organic		Effluen	Percent removal			
mg/l	Operation Days	Time Hours	Loading kg COD/day/m <sup>3</sup>	S.S. mg/l	Volatile a mg/l as CH <sub>3</sub> COO	Soluble COD H mg/l	Total COD mg/l	Soluble	BOD
Group I					.1				
1250*	7	24	0.6	22	95	68	97	94.7	92.3
2500**	7	24	1.2	48	80	84	156	96.6	93.7
5000**	10	24	2.3	92	170	290	404	94.2	91.9
8500**	8	24	4.0	95	360	562	660	93.6	92.4
Group II									
7000***	14	56	1.4	145	85	306	422	95.6	94.1

<sup>\*</sup> Phosphorus and NaHCO $_{\overline{\mathbf{3}}}$  were added.



<sup>\*\*</sup> Only NaHCO3 was added to maintain buffering capacity.

<sup>\*\*\*</sup> Raw tapioca waste.

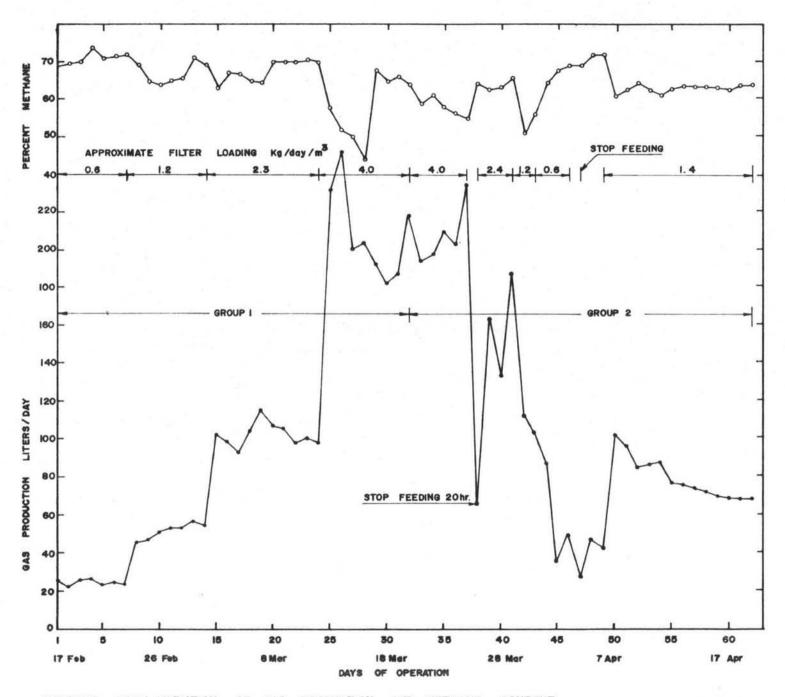
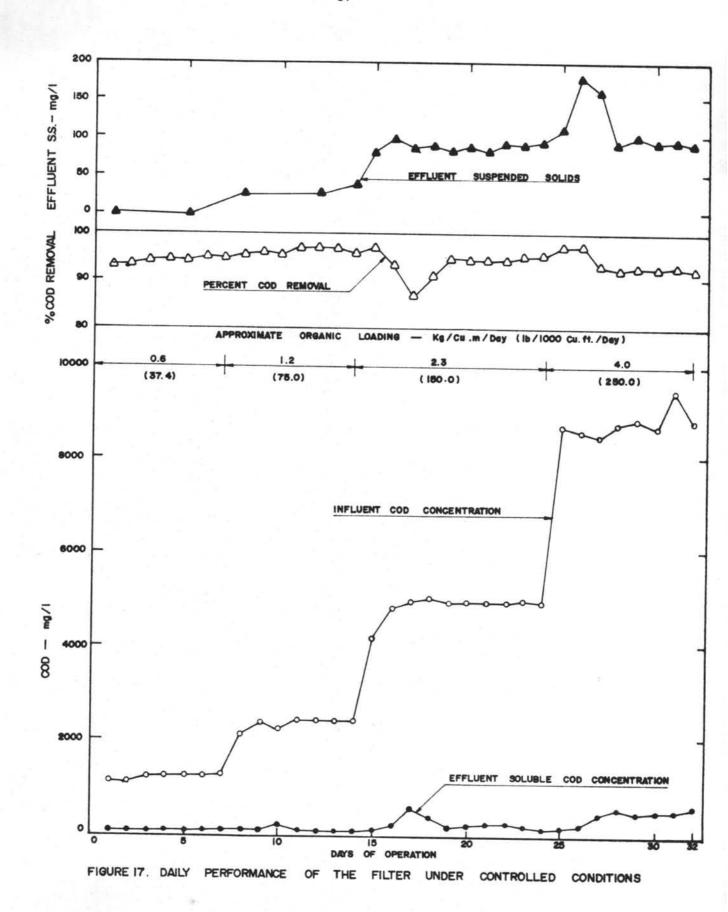


FIGURE 16 . DAILY VARIATION OF GAS PRODUCTION AND METHANE CONTENT



### 5.3.1 Effects of organic loading

5.3.1.1 Effect on COD removal Figure 18 shows correlation between steady-state removal of total and soluble COD and organic loading. Apparently, the COD removal was not sensitive to the Organic loading. The total COD removal was approximately constant at about 92 percent over a wide range of COD loading from 0.6 to 4.0 kg/cu.m./day. The soluble COD removal was about 95 percent which was slightly higher than the total COD removal. This implied that the remaining COD in the effluent was mainly in soluble and colloidal forms. It should also be noted that the filter efficiency was independent of the COD value. YOUNG and Mc CARTY (1969) found that the filter efficiency depended on the organic loading and the waste strength. The efficiency decreased with increased the loading upto a point where the dynamic balance between the acid former and the methane former were upset. For the same organic loading the efficiency seemed to increase with the increase in COD of the influent.

The results found in this study therefore, did not agree with those asserted by YOUNG and Mc CARTY (1969). The reason might be that, the bacterial population could response to organic loading as high as 4 kg COD/cu.m./day due to high synthesis rate of anaerobic bacteria when starch was used as substrate (SPEECE and Mc CARTY 1962). Therefore, the high concentration of suspended solids in the effluent was obtained at high loading (Figure 17)

5.3.1.2 Effect On gas production Figure 19 presents the variation of the gas volume and its methane content with the applied COD loading. It can be seen that the gas volume increased in direct proportion to the applied organic loading. The methane content varied slightly in a range between 64 and 70 percent. However, there was a clear trend showing that the methane content decreased with the increase in organic loading.

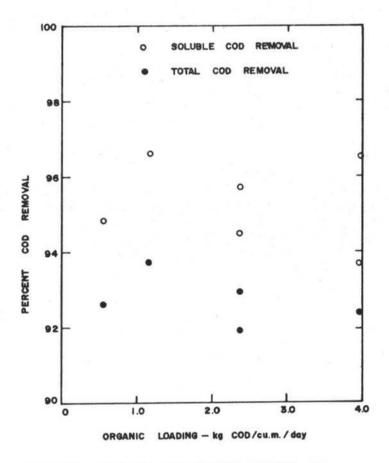


FIGURE 18 . EFFECTS OF ORGANIC LOADING ON PERCENT COD REMOVAL

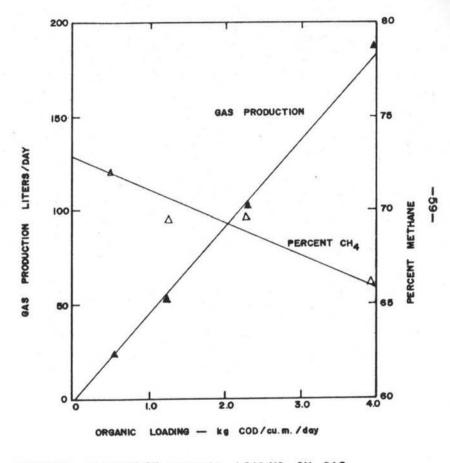


FIGURE 19 . EFFECTS OF ORGANIC LOADING ON GAS PRODUCTION

The volume of methane produced per gram of COD removed was determined and compared with the volume computed from the theoretical amount of 0.351 liter per gram COD removed as verified by Mc CARTY (1964). The results were summarized in Table 12. It can be seen that the actual methane volume was nearly equal to the theoretical volume in all cases with the maximum difference of only 8 percent. The discrepancy might be due to some methane loss with the effluent and due to the differences in pressure and temperature of the gas from standard conditions.

of filter height on COD removal, volatile acids, alkalinity and pH were similar for all organic loadings. Figure 20 shows typical results for the organic loading of 0.59 kg COD/cu.m./day. Clearly, the soluble COD removal increased rapidly with the filter height to 92 percent at 0.3-m from the bottom. In the upper 1.5-m of the filter the removal rate increased slowly to 95 percent at the filter top. This typical height v riation of COD removal was also experienced by YOUNG and Mc CARTY (1969) and was similar to the length - variation of COD removal in plugflow activated sludge aeration tank. The reason was that the filter was essentially a plug-flow reactor. This made the food-to-microorganism ratio and the rate of substrate utilization highest at the bottom and decrease with the filter height.

Figure 20bshows height variation of volatile acids, pH and alkalinity. The volatile acids concentration decreased rapidly in the lower half of the filter corresponding with the decreasing in COD value and the increase in alkalinity. Therefore, the pH was kept near 7 at all levels. The rapid removal of volatile acids and the neutral pH indicated that the acid former bacteria and the methane former bacteria were in balance.

Table 12 COD-Methane Conversion During "Steady-state"

COD COD Was	Influent Waste	A <sub>7</sub>	verage Gas Pro	Methane Equivalent	Difference from	
	Flow	Total	CH <sub>4</sub> Content	CH <sub>4</sub> Production	of COD Removed	Theoretical CH <sub>4</sub> Production
mg/l	liters/day	liters/day	percent	liters/day	liters/day	percent
1207	42	23.8	72.0	17.14	17.79	- 3.7
2406	42	54.8	69.5	38.33	35.46	+ 8.0
4708	42	105.4	70.0	73.78	69.40	+ 6.3
8178	42	187.0	66.0	123.42	128.85	- 4.2
6854	18	69.5	63.0	43.78	43.38	± o
	COD Removed mg/1 1207 2406 4708 8178	COD Waste Removed Flow  mg/l liters/day  1207 42 2406 42 4708 42 8178 42	COD Waste Removed Flow Total  mg/l liters/day liters/day  1207 42 23.8  2406 42 54.8  4708 42 105.4  8178 42 187.0	COD Removed Removed Flow         Waste Flow         Average Gas Product           mg/1         liters/day         liters/day         percent           1207         42         23.8         72.0           2406         42         54.8         69.5           4708         42         105.4         70.0           8178         42         187.0         66.0	COD Removed Flow         Waste Flow         Average Gas Production           Total         CH <sub>4</sub> Content         CH <sub>4</sub> Production           mg/l         liters/day         percent         liters/day           1207         42         23.8         72.0         17.14           2406         42         54.8         69.5         38.33           4708         42         105.4         70.0         73.78           8178         42         187.0         66.0         123.42	COD Removed         Waste Flow         Average Gas Production         Equivalent of COD Removed           mg/l         liters/day         liters/day         percent         liters/day         liters/day           1207         42         23.8         72.0         17.14         17.79           2406         42         54.8         69.5         38.33         35.46           4708         42         105.4         70.0         73.78         69.40           8178         42         187.0         66.0         123.42         128.85



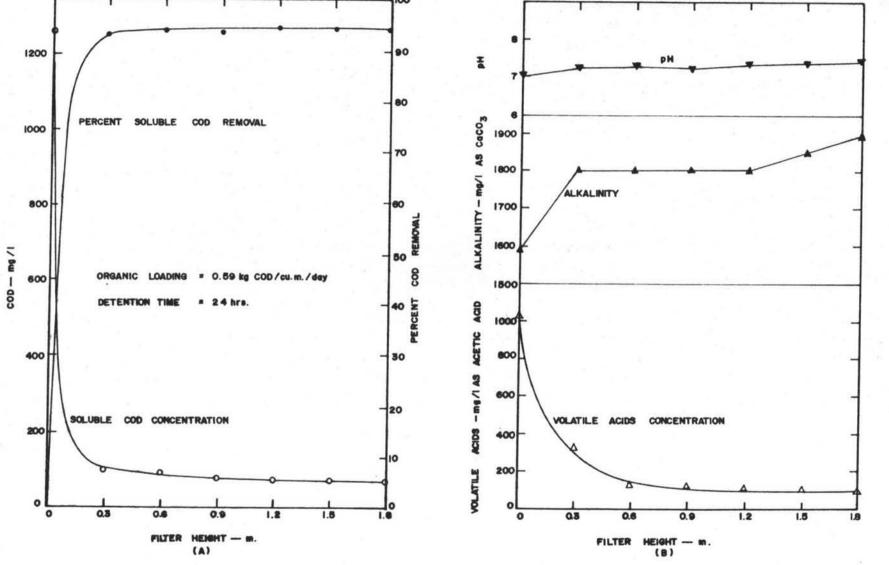


FIGURE 20 . FILTER PROFILE UNDER STEADY STATE CONTROLLED CONDITIONS

This typical pattern of COD removal implied that the first 30 cm. of the column was an active synthesis zone. In this zone the rate of COD removal and the bacterial synthesis would be highest as indicated by the presence of dense biological solids. The upper zone would merely serve as a clarification zone in which the bacterial would remain in the endogeneous respiration phase resulting in a significant rate of autooxidation. If only the first 30 cm. of the filter was considered the loading of this particular portion would be about 6 times of the total loading. At the total loading of 4 kg. COD/cu.m./day the loading of the first 30-cm. portion would be 24 kg/cu.m./day. (1,500 lb/1,000 cu.ft./day). The effective loading would be 51 kg/cu.m./day (3,185 lb/1,000 cu.ft./day), if the porosity of 0.47 was assumed. This loading was excessively high compared with the maximum loading of 200 lbs BOD/1,000 cu.ft./day normally experienced in the activated sludge process, trickling filter, and anaerobic activated sludge (STEWART 1964, GALLER and GOTAAS 1964, Mc CARTY 1964, SCHROEPFER 1959).

This finding has an important practical implication. The anaerobic filter can be designed as a high-rate process using a very high organic loading and a height of about 1.0 meter. The effluent will however, be of low quality compared to the normal anaerobic filter. It should be emphasized that shortening the filter the short-circuiting problem may be the most important problem limiting the applicability of the high-rate anaerobic filter.

- 5.3.3 Response to shock loads In the experiment the filter was subjected to shock loading when the applied loading was abruptly increased twice of the preceding value. Response of the filter to shock loading was closely studied.
- 5.3.3.1 <u>Gas production</u> Figure 16 shows daily rate of gas production and methane content of the gas at various loading through out the continuous operation. It can be seen that the rate of gas production increased almost immediately in direct proportion to the

loading. However, the methane content of the gas decreased during the first few days and increased to the original value after about 7 days. For instance, when the loading was increased from 2.3 to 4.0 kg/cu.m./day the gas production rate immediately increased from 98 liters/day to 232 liters/day, but the methane content dropped off from 70 percent to 58 percent on the first day and down to 44 percent on the forth day. It then steadily increased to 66 percent on the seventh day.

The sudden increase in the gas production rate when changing the organic loading signified that there were sufficient bacteria to cope with the increased load. At first the acid formers would rapidly break down the organic compounds producing volatile acid and the carbon-dioxide by-products. Since the methane formers would be less sensitive to the shock loading there would be an excess amount of volatile acids and carbondioxide, thus resulting in the slight drop in pH and the methane content of the gas.

5.3.3.2 Volatile acids Figure 21 shows a volatile acids profile due to the increase of COD loading from 2.3 to 4.0 kg/cu.m./day. Apparently, at any filter height the volatile acids concentration increased at first in the first few days. Then decreasing toward a new steady-state value. This pattern of variation was found to be typical for all change of loading.

The increase in volatile acids concentration indicated that the population of acid formers increased at a faster rate than the methane formers. Therefore, the volatile acids produced by the acid former accumulated until the population of methane formers was sufficient for dissimilating the excess acids. The concentration of volatile acids then decreased to an equilibrium value.

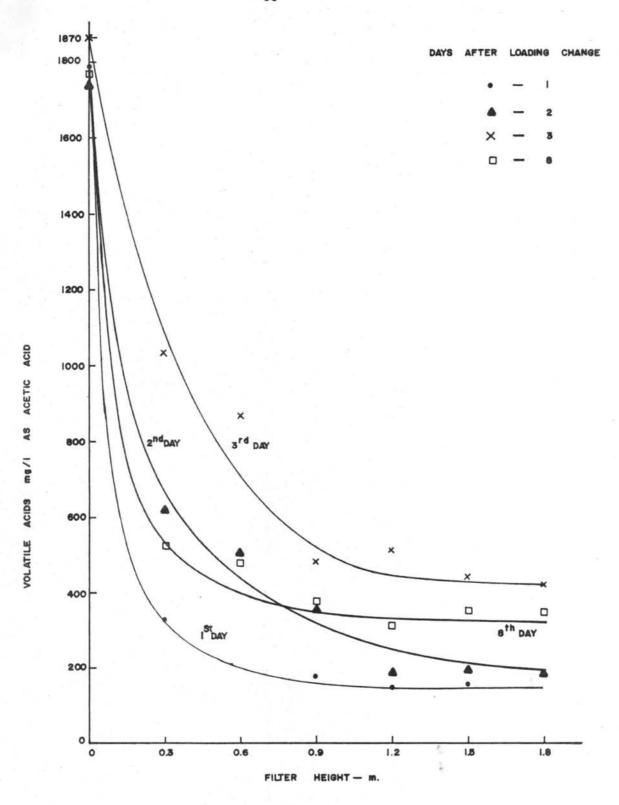


FIGURE 21. CHANGE IN VOLATILE ACIDS DURING A LOADING CHANGE FROM 2.3 TO 4.0 kg/day/m<sup>3</sup>

5.3.3.3 <u>Suspended solids</u> Figure 17 shows that the effluent suspended solids increased with the organic loading. It was found that when the loading was less then 1.2 kg/cu.m./day the effluent suspended solids concentration was never greater than 50mg/l. When the loading was increased to 4 kg/cu.m./day the effluent suspended solids concentration was relatively high, occusionally rising to 185 mg/l.

This change of suspended solids with the organic loading can be explained that at high loadings the food-to-microorganism ratio was high. As a result the bacterial growth rate was in log growth phase resulting in a low degree of bacterial auto-flocculation. At low loadings the reverse was true. This phenomenon is generally typical for most aerobic processes especially activated sludge processes.

5.3.3.4 <u>COD removal</u> It was found that the sudden increase in COD loading slighly upset the filter performance. The COD removal decreased not more than 10 percent in the first day, then increased to a new steady-state value in less than 5 days.

Figure 22 shows a COD profile of the filter after the loading was increased from 1.2 to 2.3 kg/cu.m./day. It was observed that the effect of shock loading was most significant at the filter bottom and it decreased towards the top. At 30 cm. from the bottom the COD removal dropped from 91 percent to 61 percent on the second day after increasing the loading. Then bacterial system quickly recovered resulting in the rapid increase of COD removal. On the seventh day the COD removal was as high as 92 percent. At the filter top the effect of shock loading was practically insignificant.

It can be infered that the lower part of the filter column was most sensitive to organic loading and other operational variables. Since the bacteria in the column must move in harmony the filter performance was governed by the dense biological solids in the lower part of the column.

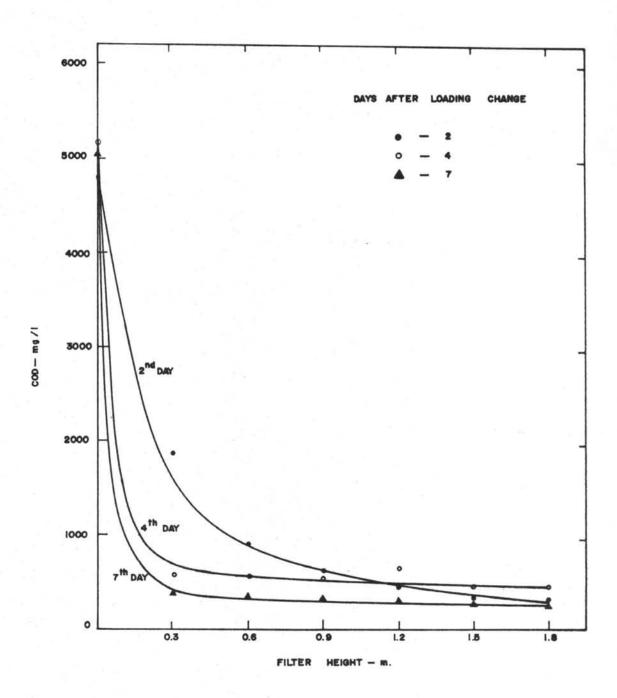


FIGURE 22 . CHANGE IN TREATMENT EFFICIENCY DURING A LOADING CHANGE FROM 1.2 TO 2.3 kg COD/cu.m./day

### 5.4 Filter Performance Under Raw Wastes Conditions

Control of pH and nutrient supplement will remarkably increase the cost of treatment. Therefore, the filter performance under raw wastes conditions was studied.

At the final stage of operation under controlled conditions the filter loading was 4.0 kg/cu.m./day. The control of influent pH was then stopped and the filter operation was continued at this loading. It was observed that though the COD removal was not significantly changed but the alkalinity rapidly decreased as shown in Figure 24. The loading was therefore, decreased to 2.4 kg/cu.m./day in an attempt to check the alkalinity drop. The loading was decreased again to 1.2 kg/cu.m./day when the alkalinity still continued to drop. But the alkalinity and pH kept on decreasing, therefore, the feed was terminated for one day. About 200 mg/l of urea was added into the influent to increase the alkalinity only for two days. The filter resumed operation at the loading of 1.4 kg/cu.m./day for 14 days. The alkalinity was observed to rise steadily. This indicated that the filter was ready for higher loadings. Unfortunately the experiment had to be terminated due to the time limitation.

The filter performance described above would have been much better if the operation commenced with low organic loadings to acclimatize the bacteria. Considering the steady rise in alkalinity and the COD removal of 96 percent, the filter would be capable of tolerating higher loading under uncontrolled conditions.

# 5.5 Effects of Shut Down

It is quite normal in industrial operation that the factory may have to be shut down for a long period to maintain and repair the machines. This will have deleterious effects on a normal biological treatment plant because the bacteria will starve. In the case of an

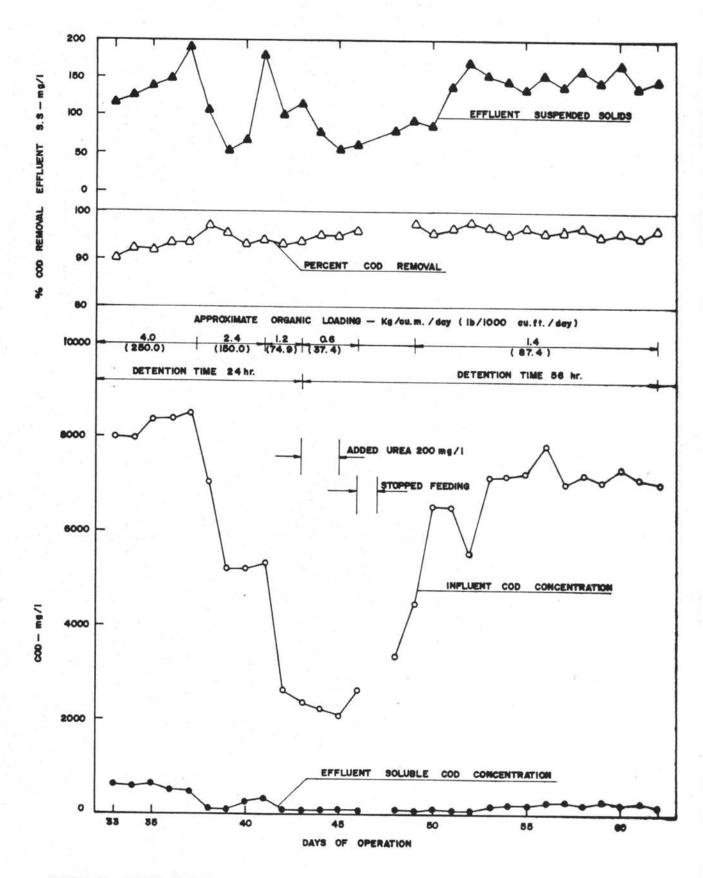


FIGURE 23. DAILY PERFORMANCE OF THE FILTER UNDER RAW WASTE CONDITIONS



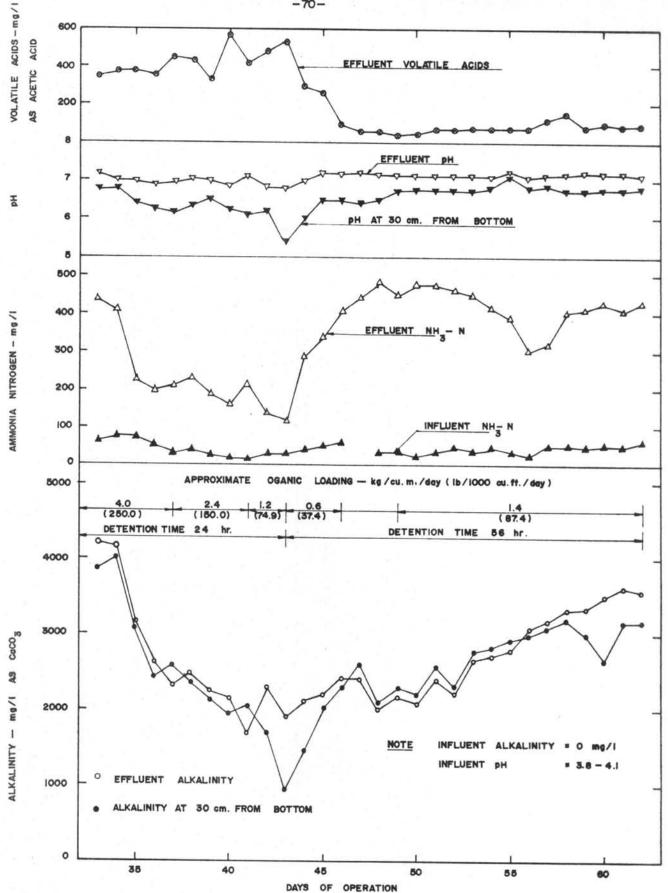


FIGURE 24. DAILY PERFORMANCE OF THE FILTER UNDER RAW WASTE CONDITIONS

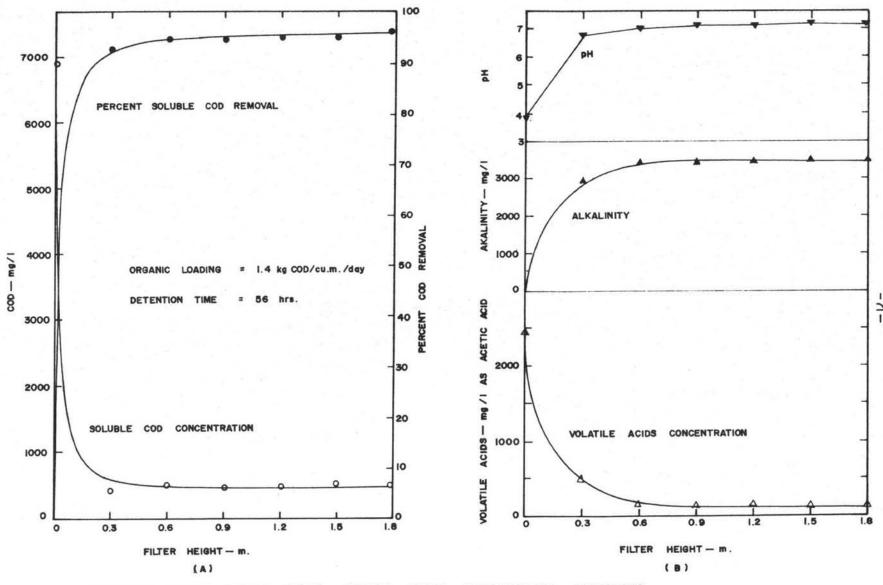


FIGURE 25. FILTER PROFILE UNDER STEADY STATE UNCONTROLLED CONDITIONS

activated sludge plant the starving microorganism will oxidize themselves resulting in degeneration of the flocs. When the factory resumes operation the waste treatment plant will have to be start-up again.

To simulate the effect of the factory shut-down, the filter feeding was terminated for 15 days after 62 days of continuous operation. Then the filter was fed with a raw tapioca starch waste having a COD corcentration of 5000 mg/l. The feed rate was 13 liters/day and the organic loading was 1.0 kg COD/cu.m./day. Figure 26 & 27. Shows the daily recorded values of COD profile, alkalinity profile, the rate of gas production, methane content of the gas, influent COD, effuent COD, influent pH and effluent pH. It was observed that gas production commenced immediately after starting feeding. The gas production rate rapidly increased reaching the steady-state rate within 5 days. The methane content of the gas was relatively high during the first few days because carbondioxide was used by the methane bacteria as a hydrogen acceptor in the methane synthesis. It was interesting to note that the effluent pH remained close to 7 at all time though the influent pH was constant at about 3.9.

Figure 26 shows COD and alkalinity profile of anaerobic filter for various days after starting feeding. Apparently, the COD at any height was virtually constant. The greatest COD removal was observed in the first 30 cm. from the bottom.

When the filter operation was stopped for 15 days the remaining ammonia would combine with carbondioxide yielding ammonium bicarbonate. Since there was no production of volatile acids during this period the alkalinity accumulated to 3,600 mg/l at all levels. When the feeding was resumed the alkalinity would be neutralized by the volatile acids. The drop in alkalinity with time could be clearly observed at all levels. On the sixth day the alkalinity was about 1,800 mg/l which would be at equilibrium. In spite of the decrease in alkalinity, the ratio

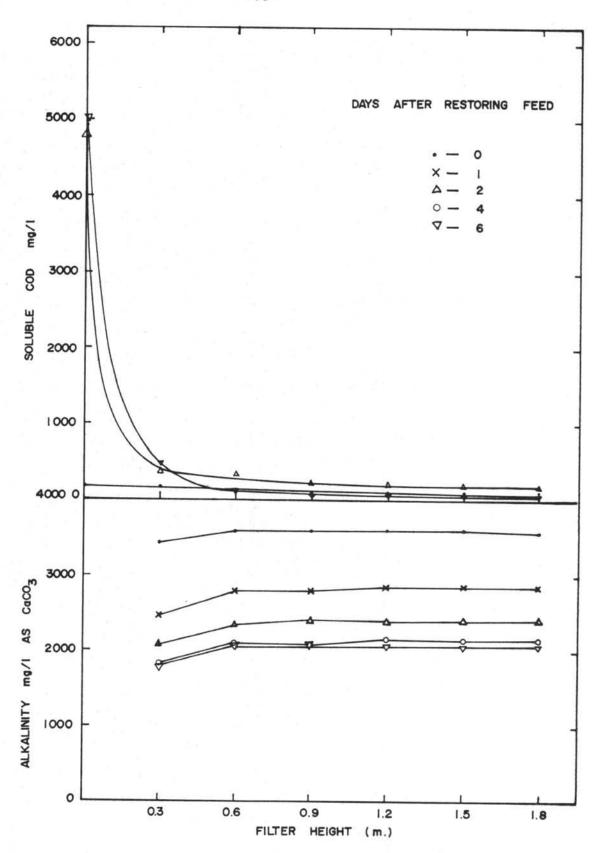


FIGURE 26. FILTER PROFILE RECEIVING 5000 mg/I COD AFTER STOP FEEDING FJK



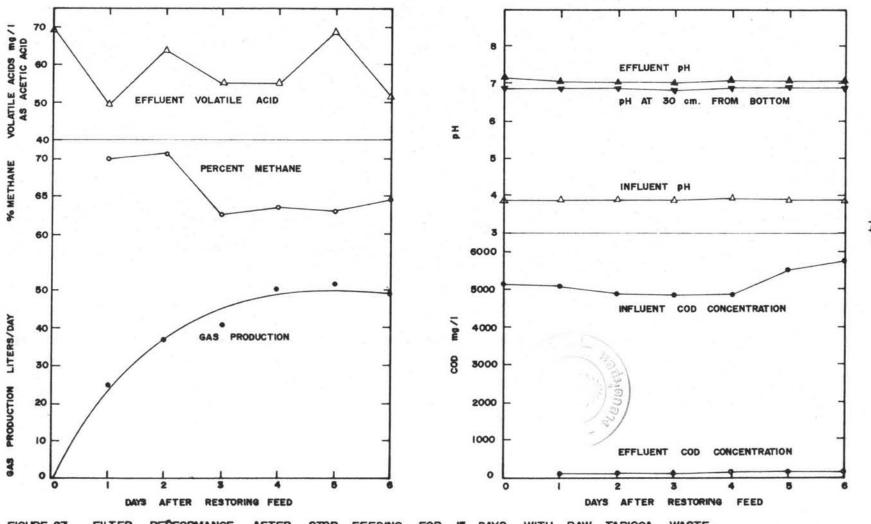


FIGURE 27 . FILTER PERFORMANCE AFTER STOP FEEDING FOR 15 DAYS, WITH RAW TAPIOCA WASTE, COD LOADING 1.0 kg/cu.m./day

between the volatile acids and the alkalinity was always less than 0.3 keeping the pH in the filter neutral at all time.

The result of experiment led to a conclusion that the anaerobic filter could quickly regain its efficiency even it was shut down for 15 days.

#### 5.6 Biological Solids

Visual observation of the biological solids within the filter revealed that they were not present as slimes firmly attached to the stone surfaces as in trickling filter. The solids were essentially large flocs loosely located in the interstitial spaces. The flocs were so heavy that they were not easily carried upwards by the rising gas bubbles. Although some of the flocs might be lifted by the gas bubbles, but the buoyant solids would hit the overlying stones and settle back almost to their original positions. Therefore, the gas bubbles would be free to rise. However, at the upper part of the filter column some of the biological solids might be entrained with the effluent. The suspended solids at various depth are whow in Table 13.

The limited time available for this research did not allow a thorough investigation into the kinetics of the bacterial growth in the filter. This topic is recommended for future study.

# 5.7 Microbiological Observation

All samples withdrawn from the filter were subjected to routine microscopic examination. A predominant filamentous growth was usually observed. YOUNG and Mc CARTY (1969) also found this type of growth in their study, according to their belief was a characteristics of Methanobacterium sohngenii. Close microscopic examination revealed that the filaments were composed of chains of

Table 13Suspended Solids in Samples Withdrawn Through the Sample Ports of Filter.

Days of		Suspended Solids (mg/l) at Varions Depth from Bottom						
Operation 0.3-m	0.3-m	0.6-m	0.9-m	1.2-m	1.5-m	1.8-m		
0	142	85	36	20	0	0		
10	185	198	180	72	60	32		
20	580	287	250	162	80	72		
30	728	364	308	264	152	144		
40	1300	940	790	540	276	184		
50	1870	1250	630	364	336	88		
60	1460	840	510	210	220	145		

short rod-shaped forms. In the upper part of the filter some ciliated protozoa was also found.