Chapter 4

Material and Methods

4.1 Experimental Equipment

A flow diagram of the UASB pilot-scale experiment is shown in Fig. 4.1. The equalizing tank installed provided a storage facility enabling the experiments to be carried out over a full 24-h period. The plant was provided with a flow control box enabling application of flow from 0.2-4.0 l/m (0-1 gal/m). The two centrifugal pumps were alternately used to pump the waste into the single influent port with a cone shape, forcing the wastewater upward through the sludge bed present in the lower part of the reactor. The reactor was made of stainless steel, had an internal diameter of 30 cm, and was 4.7 m in height. The total volume was 346.8 litres but the effective volume was 314.5 litres. Ten sample ports were constructed at vertical intervals of 40 cm, mounted in the reactor wall, with the first port 25 cm from the bottom. No mechanical mixing or sludge recirculation was applied for the sake of sludge settleability.

The reactor was equipped inside the upper part with a proper gas-liquid-solid separator or settler (more detail will be mentioned further). When the mixed liquor arrived at this settler, the sludge particles were settled out and returned to the digester compartment, whereas the effluent passed through a siphon tube that separated

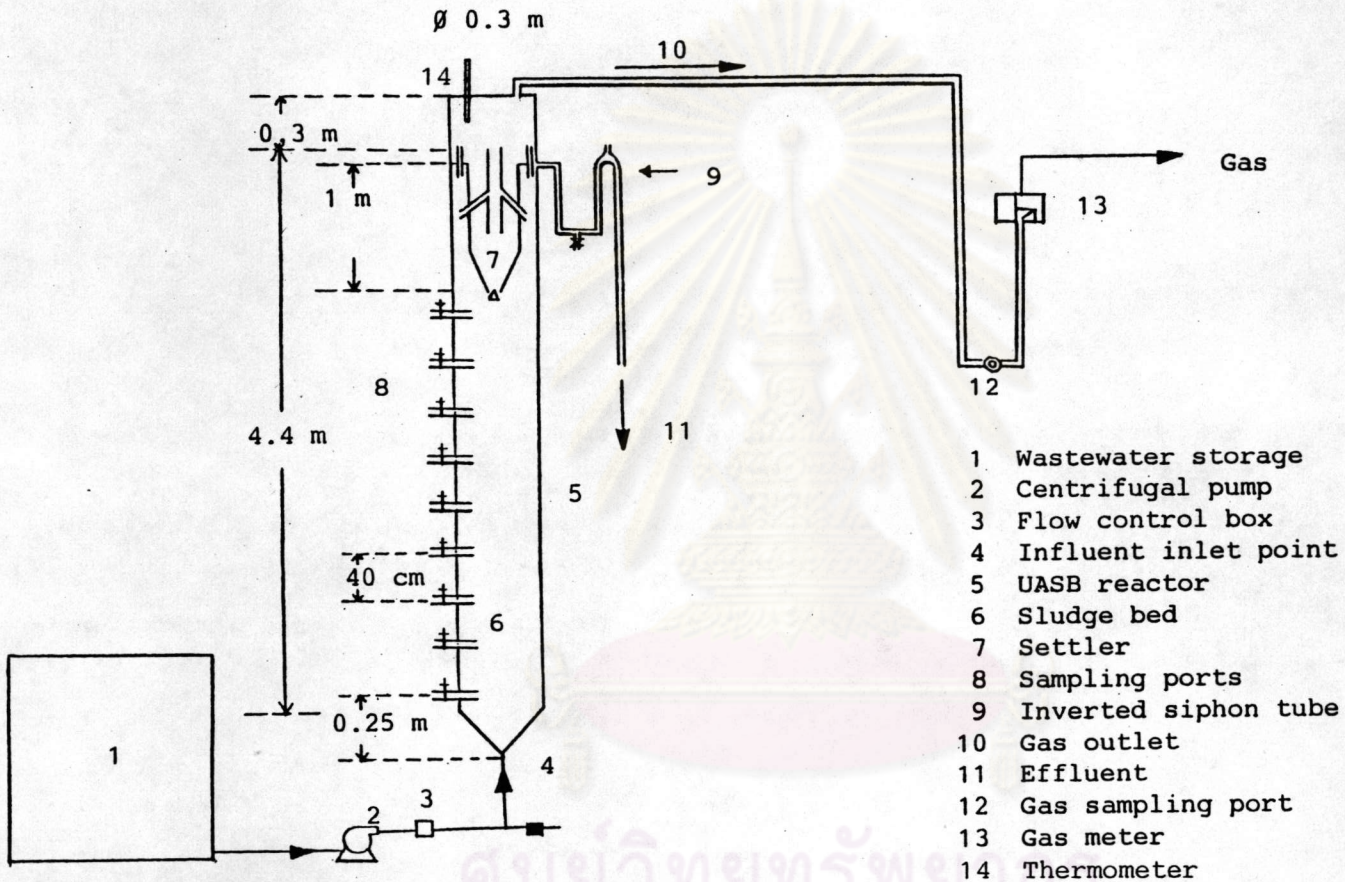

Fig.4.1 Schematic diagram of the pilot-scale UASB experiment

the gas produced from the liquid.

The general designation of this internal settler was based on the guidelines provided by Lettinga (as described in Chapter 2). As for the hydraulic surface overflow rate of the settler used in this experiment, it was selected to fix at 1.0 $m^3/m^2/hr$. Due to that hydraulic surface overflow rate was calculated on the basis of hydraulic flow rate/total surface area of the settler, the different setters with inner diameters of 14.2, 19.3, 23.2, 26.5 and 32.2 were used, together with the HRT's of 24, 12, 8, 6 and 4 hr, respectively. They all were made of zinc with a height of 1 m and an inclined wall of approximately 50° slope.

4.2 Substrate

Brewery wastewater, after passing through a 1-mm. square mesh screen, was daily collected from the main wastewater pipe of the Boon Rawd Brewery Treatment Plant. The most important characteristics of raw brewery wastewater are presented in Table 4.1.

It can be seen that the wastewater is a low-strength type waste which has sufficient levels of nutrients for anaerobic bacterial growth. The ratio of BOD to COD is between 0.6-0.7. The wastewater has a widely fluctuating COD during the time of day and the days of week. It was observed that the COD influent was lower than the mean COD value of 2175 g/m^3, from Saturday until Tuesday, after that the COD value was increased again. Thus, the brewery wastewater used in this study, except for the first start-up period, was prepared in batches to keep the COD concentration stably at the

Table 4.1

The characteristic of Brewery Wastewater (data was obtained from the Boon Rawd Brewery Wastewater Treatment Plant, Bangkok, in 1984).

Parameter		Range	Mean
COD	g/m^3	350 - 4000	2175
BOD_5	g/m^3	240 - 2640	1440
TS	g/m^3	1678 - 3286	2482
SS	g/m^3	280 - 556	418
TKN	g/m^3	15.0 - 60.0	37.5
TPO_4^{3-} as P	g/m^3	2.8 - 10.0	6.4
pH		4.6 - 11.0	-
Temperature °C		35 - 38	-

mean value (approximately 2000 g/m^3). When the wastewater COD concentration was low, the clarified beer wort[4] solution whose exact COD value has already been known, was added to the wastewater, but when the COD concentration was high the tap water was used to dilute the wastewater. Na_2CO_3 solutions were added for a buffer capacity in the wastewater over the entire experimental period. Its characteristics were summarized in Table 4.2.

4. See the preparation of beer wort in brewing process in Chapter 3. COD value of clarified beer wort solution is 205,882 g/m3

Table 4.2

Characteristic of raw (screened) wastewater

Parameter		Range	Mean
COD total	g/m^3	408 - 4192	2177
COD filtered	"	325 - 3677	1757
BOD_5	"	325 - 2875	1562
TS	"	1184 - 5592	2950
SS	"	82 - 1690	386
VSS	"	38 - 697	208
TKN	"	12.6 - 91.0	31.1
TPO_4^{3-} as P	"	1.8 - 12.5	5.4
pH (raw)		3.6 - 9.2	6.2
(buffered)		6.7 - 10.2	8.2
Total Alkalinity	g/m^3 as $CaCO_3$	631 - 1082	799.4
VFA	g/m^3 as HAc.	80 - 1350	440.0
Temp. at sludge bed	°C	34 - 42	38.0

From Table 4.2, the volatile suspended solids was only 7% of total solid which indicated a very low fraction organic matter in the form of suspended solids. Consequently, soluble organic existing in the wastewater might be amendable to anaerobic process. The ratio of COD:N:P of 100:1.46:0.26 indicated that there were adequate nutrients necessary for cell growth requirement.

4.3 Seed Sludge

Digested sewage sludge taken from the bottom of the equalizing tank at the Boon Rawd Brewery Wastewater Treatment Plant was used as the seeding material.

4.4 Sampling and Analyses

Influent and effluent samples taken for chemical analyses were grab sampled from sampling ports everyday from Monday to Saturday; although on Sunday the reactor was continuously fed, but no sample was taken.

The following analyses were performed :

Influent - Effluent

pH	daily on spot
COD_t, COD_f	daily grab sample
BOD_5	" "
TS	" "
SS, VSS	" "
Alkalinity	" "
Volatile Fatty Acid (VFA)	" "
Total kjadahl Nitrogen (TKN)	" "
Total Phosphate (TPO_4^{3-})as P	" "

Gas

Production	daily on spot
Composition	" "
Temperature	" "

Sludge Bed

Temperature	daily on spot

Profile

COD_f	after ending of each run
SS, VSS	" "
Alkalinity	" "
VFA	" "
pH	" "

For analyses on the influent and effluent, the Standard Methods for the Examination of Water and Wastewater (1980) were followed. The total volatile fatty acid (VFA) content and alkalinity were measured by direct titration according to DiLallo and Albertson (1961). Gas production was measured by using gas meter designed by Opaswatchai (1984) (see Fig. 4.2). Gas composition was immediately analysed for the CH_4 content by Orsat gas analyser (Fig. 4.3), but also certain amounts of gas samples were taken with a 50-ml. syringe and analysed for confirmation by using a Shimadzu gas chromatograph, model GC-7AG, equipped with two column-MS-5A and PQS TCD; column temperature was 70°C and injection temperature was 90°C.

The resulting comparison test between the two different methods of gas analysis is shown in Table 4.3 and indicates that they are not significantly different at 5% level of confidence.

Table 4.3

Comparative data of CH_4 gas analysed by GC and Orsat absorption method.

Date	%CH_4 by GC	%CH_4 by ORSAT	Differential (d_i)
1-2-89	78.6	77.0	1.6
8-2-89	81.85	79.6	2.25
15-2-89	74.74	73.0	1.74
22-2-89	73.05	74.4	-1.35
1-3-89	65.55	67.0	-1.45
8-3-89	78.77	80.4	-1.63
15-3-89	69.36	70.0	-0.64
22-3-89	81.43	83.0	-1.57
29-3-89	75.77	78.0	-2.23

$H_o : \mu_d = 0$

$H_1 : \mu_d \neq 0$

$n = 9$

$\bar{d} = -0.3644$

$S_{\bar{d}} = 0.5760$

$Sd = 1.7279$

$\therefore t_{cal} = -0.6326$

But at $\alpha = 0.05$, $t_{Table} = t_{0.025,8} = \pm 2.306$

$\therefore$ Accept Ho

There is no significant different between the two methods at 5% ($\alpha = 0.05$) level of confidence. Therefore the Orsat absorption method is accepted to be used for CH_4 testing in this study.

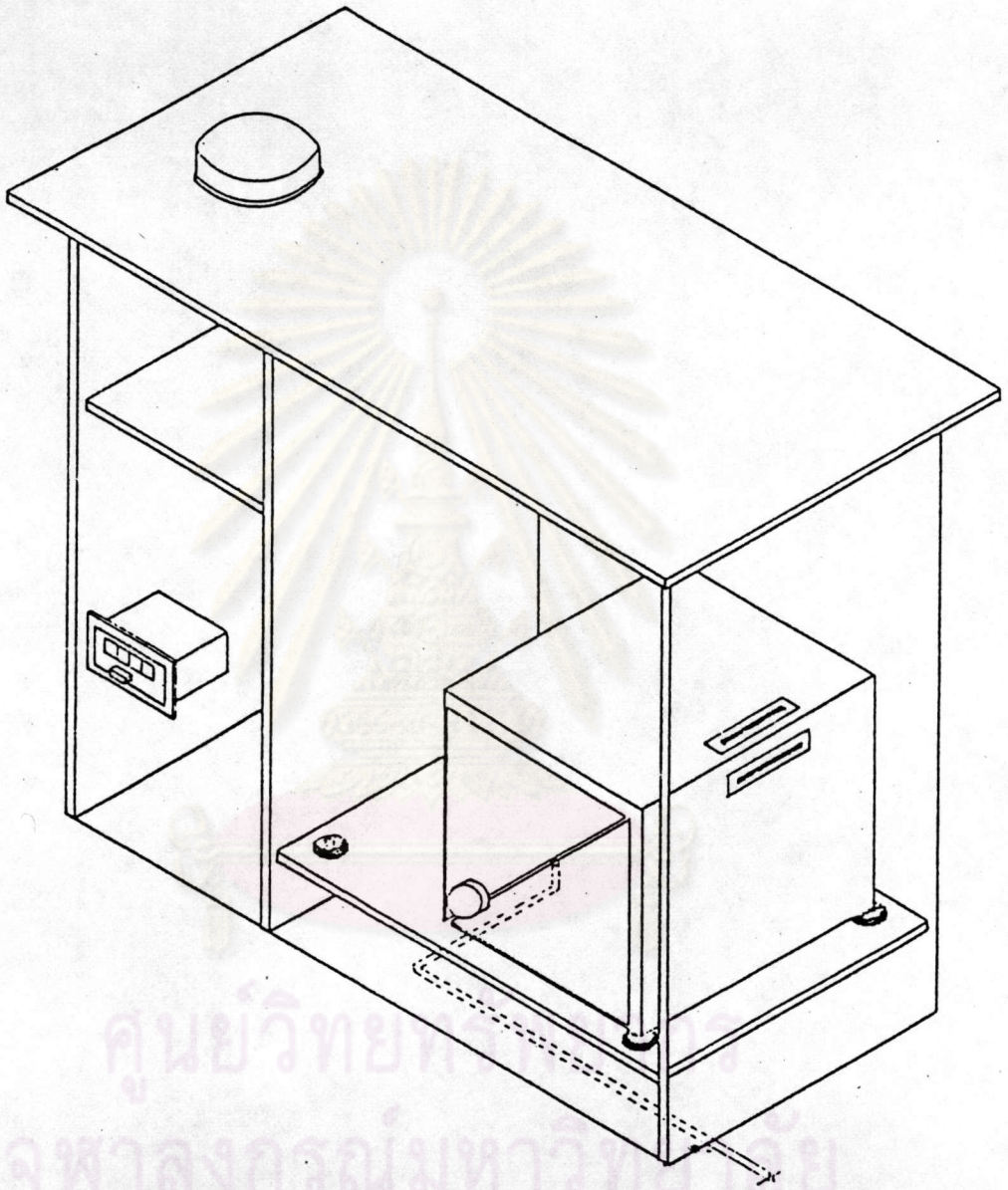

Fig.4.2 Gas meter (Opaswatchai, 1985)

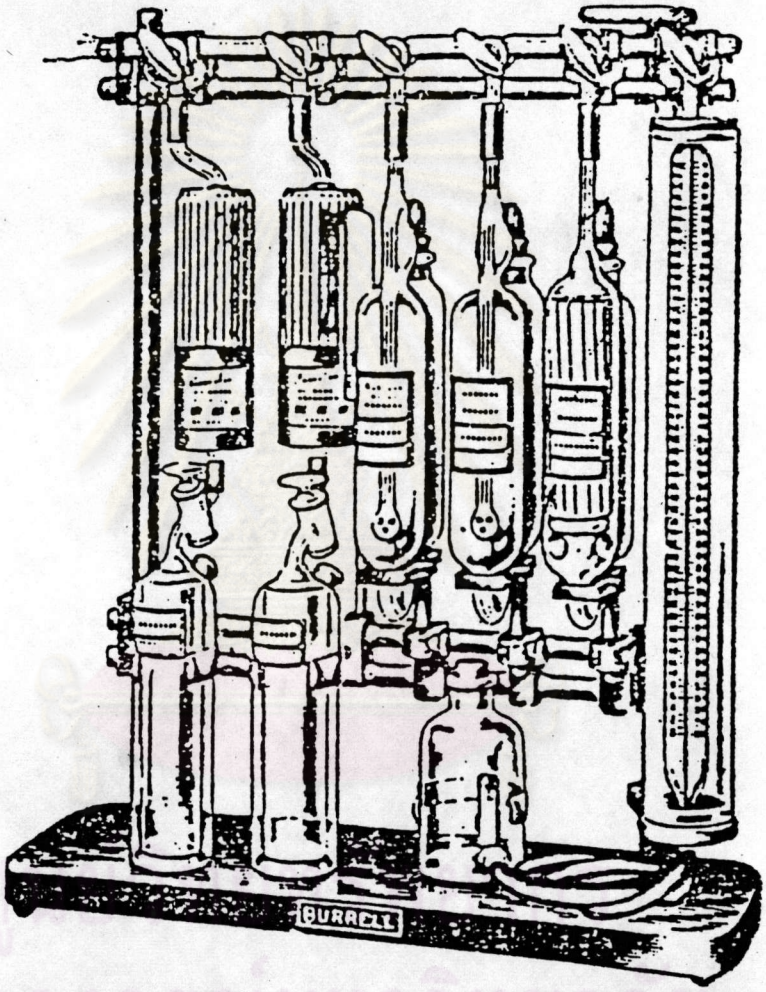

Fig.4.3 Orsat gas analysis apparatus (Sawyer, 1978).

Some granular sludge observations were made with a scanning electron microscope (SEM) by means of a JEOL JSM-T220A Scanning Microscope. Both gas chromatography and electron microscope were used to analyse the samples at the Scientific and Technological Research Equipment Center, Chulalongkorn University.

4.5 Experimental Procedure

4.5.1 The First Start-up

In the first part of the experiment, the main goal of the start-up was exclusively to cultivate the granular sludge in the UASB pilot-scale reactor. The reactor was started up by filling it with the digested sewage sludge of an initial amount of approximately 17-g suspended solid (SS)/l and feeding it with the feeding solution prepared by mixing clear beer wort surplus yeast[5] with tap water (hardness 7° CaO or 125 mg/l as $CaCO_3$) at a ratio of 1:300. This results in an approximately 1000 g/m^3 COD feeding solution. The influent pH was kept near neutral by employing soda-ash (Na_2CO_3) as a basic material for pH control. The supplement of calcium is not necessary because of a fairly amount of hardness existing in the tap water used. This feeding solution was fed with a centrifugal pump for continuously feeding experiments.

4.5.2 The Reactor Performance Test

The second part of the experiment was performed with HRT variations of 24, 12, 8, 6, and 4 hours, respectively, while

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5. COD value of clear beer wort surplus yeast is 338,000 g/m3

the hydraulic surface overflow rate of the settlers was fixed at 1.0 $m^3/m^2/hr$. The brewery wastewater was collected in the storage tank and continuously fed into the bottom of the reactor after being prepared with the COD concentration of approximately 2000 g/m^3. The reactor performance was routinely assessed by daily determination in the laboratory during the interval of each HRT (from the beginning of run to the end of the steady state).