

**ANAEROBIC DIGESTION OF BAKER'S YEAST WASTEWATER  
USING A UASB REACTOR AND A HYBRID UASB REACTOR**

By

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## ABSTRACT

The start-up and step-up operation of two 16-liter, continuously operated, upflow anaerobic reactors receiving baker's yeast wastewater is presented in this thesis. The two reactors (A and B) were almost identical in construction. Reactor A was a conventional upflow anaerobic sludge blanket (UASB) reactor, and reactor B was a hybrid reactor. In addition to all the features of a UASB reactor, a fixed-film structure was installed in the mid section of the reactor B. Both reactors were operated at 35°C and at a constant hydraulic retention time of 7 days. The waste strength, expressed in chemical oxygen demand (COD), was varied from 8 g COD liter<sup>-1</sup> (during the start-up) to 58 g COD liter<sup>-1</sup>. The organic loading rate ranged from 1.1 to 9.4 g COD liter<sup>-1</sup> day<sup>-1</sup>. The start-up lasted for the first 46 days. Towards the end of the start-up, methane production rates of 0.23 and 0.28 liter CH<sub>4</sub> liter<sup>-1</sup> day<sup>-1</sup> and COD reductions of 62.2% and 67.2% were achieved at organic loading rates of 1.1 and 1.3 g COD liter<sup>-1</sup> day<sup>-1</sup> for reactors A and B respectively. During the step-up operation, maximum methane production rates were, for reactors A and B respectively, 0.91 and 0.95 liter CH<sub>4</sub> liter<sup>-1</sup> day<sup>-1</sup> at organic loading rates of 5.8 and 6.4 g COD liter<sup>-1</sup> day<sup>-1</sup>. In addition, reactor profiles for sludge concentration, pH, volatile fatty acids, and COD are also presented.

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## LIST OF ABBREVIATIONS

BOD <sub>5</sub>	Five-day biochemical oxygen demand according to the <i>Standard methods for the examination for waste and wastewater</i>
COD	Chemical oxygen demand
DS	Dry solids
TS	Total solids
TSS	Total suspended solids
TVFA	Total volatile fatty acid
UASB	Upflow anaerobic sludge blanket
VFA	Volatile fatty acid
VS	Volatile solids
VSS	Volatile suspended solids

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# I. INTRODUCTION

## A. Problem Statement

Baker's yeast wastewater is a by-product of the bakery yeast growing process. The wastewater consists mostly of polysaccharides, long-chain organics, minerals, and sulfate. The wastewater is characterized by a high COD content (60 to 100 g liter<sup>-1</sup>), which prohibits its direct disposal into the sewer. Aerobic treatment of this wastewater is not possible without substantial dilution, since aerobic microorganisms can only degrade wastewaters with COD concentrations in the order of a few thousand milligrams per liter. Anaerobic digestion, however, can treat wastewaters of high organic strengths, requires little energy to operate, and yields methane which may be burned as a supplemental energy source.

The presence of high concentrations of sulfate in the wastewater causes concern in relation to anaerobic fermentation. High concentrations of sulfurous compounds have been known to either lower the anaerobic digestion efficiency<sup>1</sup> or, in worse cases, hamper the process altogether. Nevertheless, the problem of sulfate toxicity is not impossible to overcome.

## B. Proposed Solution and Objectives of the Experiment

The process chosen was anaerobic digestion, and the type of reactor was the upflow anaerobic sludge blanket reactor. Two reactors operated continuously were used in this experiment. Reactor A was a conventional UASB reactor; reactor B was a hybrid UASB reactor since, in addition to the basic UASB configuration, it was equipped with ropes as a fixed-film medium to increase surface area for bacteria to attach. UASB reactors were employed in this experiment because they are efficient and yet simple in design, and as a result have been used extensively in Europe and

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<sup>1</sup> The digestion efficiency technically refers to methane yield, which is the amount of methane gas produced per unit of feed (e.g., a gram) utilized.

China (Wang *et al.*, 1985). The success of UASB reactors lies in their ability to retain sludge, which is essential to high reactor performance.

The objectives of this experiment are

- 1) To investigate the treatment efficiencies of a UASB reactor (reactor A) and a hybrid UASB reactor (reactor B) fed with baker's yeast wastewater.
- 2) To study the behaviors of the two reactors during the start-up and step-up operation.
- 3) To determine the impact of the fixed-film system on the performance of reactor B, as compared with a conventional UASB reactor and other reactors.
- 4) To gain some insight into the extent of sulfate toxicity effects on the performances of the two reactors.

### **C. Thesis Organization**

This thesis is organized into chapters: "INTRODUCTION," "LITERATURE REVIEW," "MATERIALS AND METHODS," "START-UP," "STEP-UP OPERATION," "CONCLUSIONS," "RECOMMENDATIONS," and "REFERENCES". The "INTRODUCTION" serves as the overall introduction for the thesis. The "MATERIALS AND METHODS" is also a general section which explains such items as how the reactors used in the experiment functioned; seed sludge employed; feed material; operational regime; and chemical analyses conducted. Instead of lumping everything together in one single results-and-discussions chapter, the results and discussions have been treated in two separate chapters, namely, "START-UP" and "STEP-UP OPERATION". The latter chapter is divided into two sections: "Steady-state and Transient Analysis" and "Reactor Profile Study". In particular, the reason for treating the steady-state and transient results and discussions in the same chapter is merely a matter of compactness. Had the steady-state and transient parts been dealt with individually, repetition would be unavoidable.

Because the chapters "START-UP" and "STEP-UP OPERATION" are rather extensive, a chapter conclusion has been included for each of them. The thesis-wide conclusion then sums up all of the chapter and sectional conclusions, and is followed by the recommendations based on the thesis results and findings.

## II. LITERATURE REVIEW

### A. Anaerobic Wastewater Treatment

#### 1) Overview

With growing environmental concerns and energy considerations, anaerobic treatment of wastewaters has drawn substantial interest (Lettinga *et al.*, 1980). Listed below are the advantages and disadvantages of anaerobic treatment of wastewaters (Lettinga *et al.*, 1980):

#### Advantages

- No energy is required for aeration.
- Nutrient requirements are low.
- Methane is generated as a by-product, which may be burned as a supplemental energy source.
- High loading rates can be accommodated under favorable conditions.
- Excess sludge production is low.
- The produced sludge is highly stable and dewatered readily.
- Active anaerobic sludge may be preserved unfed for a long period of time.

#### Disadvantages

- Anaerobic bacteria grow slowly, so long periods of time are required for reactor start-up, and recovery from upsets.
- Anaerobic digestion is very sensitive to environmental and operating factors such as toxic compounds and pH.
- Anaerobic treatment is only a front-end treatment process; post-treatment of the treated wastewater is needed to meet disposal requirements.
- More experience needs to be gathered with the application of the process to direct treatment of wastewaters.

## 2) Anaerobic Digestion

Anaerobic digestion involves four stages: hydrolysis, acidogenesis, acetogenesis, and methanogenesis (Chung *et al.*, 1988; Hill *et al.*, 1987). In hydrolysis, complex organic matter in wastewater is broken down to soluble organics; next, in acidogenesis, they are further broken down, by acidogenic bacteria, to fatty acids (acetic, propionic, and butyric acids). Once the organic matter is in the form of acetic acid, it is then converted to methane and CO<sub>2</sub> by methanogenic bacteria (methanogenesis). Long-chain volatile fatty acids (VFA's), propionic and butyric acids, must first be degraded to acetic acid, H<sub>2</sub>, and CO<sub>2</sub> by hydrogen-producing bacteria (acetogenesis). Alternatively, another group of bacteria (homoacetogenic bacteria) also contribute to methanogenesis by converting H<sub>2</sub> and CO<sub>2</sub> to methane. According to Chung *et al.* (1988) and Hill *et al.* (1987), it is well accepted that 70% of the total methane derives from acetic acid while the remaining 30% results from the conversion of H<sub>2</sub> and CO<sub>2</sub>. Methanogenic bacteria are known to grow more slowly than acidogenic bacteria; besides, methanogenic bacteria are rather sensitive to environmental factors such as pH and inhibitory compounds. Hence, it is important to make sure that the methanogenic bacteria are not under stress if the digestion process is to proceed stably.

Based on the aforementioned criteria, an accumulation of acetic acid implies that methanogens are not able to convert acetic acid into methane as fast as it is produced. Furthermore, an increase in propionic-to-acetic ratio (P/A ratio) indicates that the bacteria that break down propionic acid to acetic acid are under stress, since it has been found that propionic acid is the precursor to acetic acid (Hill *et al.*, 1987; Segretain *et al.*, 1987; Denac *et al.*, 1988).

There has been much effort put into establishing criteria which give some indication as to how healthy an anaerobic digester is and when process failure is imminent. Hill *et al.* (1987) conducted a literature survey on the acetic and propionic

acids and their ratio as an indicator of process stability. Later, Hill *et al.* (1988) went on with further work, focusing on the longer-chain VFA's such as butyric and valeric acids. Their most important conclusions are as follows:

- 1) Total volatile fatty acid (TVFA) concentrations greater than 2000 mg liter<sup>-1</sup>, acetic acid concentrations higher than 800 mg liter<sup>-1</sup>, and a P/A ratio greater than 1.4 are reliable methods of predicting the failure of anaerobic process. Of these criteria, the combination of acetic acid and the P/A ratio provides the best index for digestion failure prediction.
- 2) Longer-chain VFA's, butyric and valeric acids and especially their iso-forms play an important role in detecting the stress which precedes failure.
- 3) When the iso-butyric and iso-valeric acids concentrations are below 5 mg liter<sup>-1</sup>, the anaerobic digestion is under little or no stress. If these iso-forms rise to the concentration range of 5-15 mg liter<sup>-1</sup>, the digester is under much stress and failure is impending if the problem is not corrected. Concentrations of iso-forms higher than 15 mg liter<sup>-1</sup> means that failure has already set in.

## **B. UASB Concept**

The basic idea of the UASB process, first conceived in 1971 by Lettinga and his co-workers in The Netherlands, is the reactor's ability to retain a high concentration of viable sludge, combined with a good contact between the sludge and the incoming wastewater (Lettinga *et al.*, 1980). The wastewater is introduced into the reactor from the base. It then slowly flows up the reactor column through the sludge bed and blanket and is digested in the process.

The ability to keep active sludge in the reactor is vital both to a quick start-up and superior reactor performance. The UASB reactor achieves high sludge retention through a liquid-solids-gas separator. This separator comprises a cone, with its larger base either facing up or down, sealed to the reactor inner wall near the top of the reactor. In either case, the underlying principle is the same: the device achieves high sludge retention by creating a quiescent zone near the top of the reactor which helps

rising sludge flocs form larger aggregates and sink back down. The mechanism through which sludge flocs rise can be turbulence or flotation. The type of separator discussed here is one with the cone's base facing upward. At some clearance directly in front of the cone tip (which faces downward) is fitted a smaller cone with its tip pointing toward the tip of the larger cone. This construction minimizes sludge loss by blocking the uprising mixed liquor from hitting directly the tip of the larger cone. Generated biogas, though, becomes collected in the smaller cone, and, when it fills the smaller cone, it escapes from the perimeter of the cone upward and collects in the space between the reactor liquid surface and the base of the larger cone which is sealed to the reactor wall. The biogas collecting in the cone headspace is then routed to a gas volume measuring device. In addition, as flocs of bacteria rise, through buoyancy provided by rising gas bubbles or lack of settleability of the bacteria, and travel through the cone, the liquor velocity tapers off due to the enlargement of the diameter of the cone. As a consequence, the rising bacteria particles slow down and tend to gather near the liquor surface and form larger flocs which will then sink down the reactor.

### **C. Sludge Granulation in the UASB Reactor**

The success of a UASB reactor depends on the development and immobilization of a sludge with high activity and superior settling characteristics (Lettinga *et al.*, 1980, 1985). Newly generated bacteria may become immobilized on particulate matter and immobile surface through mechanisms of attachment, entrapment, or intertwining. In the case of sludge immobilization on particulate matter, aggregation of bacteria takes place and leads to the formation of granules. In the case of immobilization onto a fixed medium, a layer of biomass develops on the surface of the fixed-medium. Fixed-film immobilization, in particular, plays a significant role in

retaining sludge, especially in reactors started up with thin and not-so-well-settled seed sludge. The idea of retaining as much biomass as possible is applicable for all anaerobic reactors treating a whole range of wastewaters.

Lo *et al.* (1984), treating dairy manure in laboratory-scale 4-liter batch reactors, confirmed the significance of support material for retaining biomass and thus improving treatment efficiency and tolerance to stress. It has been found that immobilization both on fixed surface and on particulate matter occurs rather easily. The basic mechanism for granulation is briefly discussed here. Newly generated bacteria attach to the primary nuclei which are the heavier sludge flocs in the seed sludge, which is commonly digested sewage sludge. Later in the start-up, the eruption of attached bacterial films or break-up of larger bacterial aggregates will give rise to secondary nuclei. Similar to the primary nuclei, the secondary nuclei themselves will eventually form granules. Unlike a fluidized-bed reactor, for instance, the UASB reactor has the ability of retaining the smaller secondary nuclei, which can gradually grow into larger aggregates (Lettinga *et al.*, 1985). The sludge with these qualities is one that is well-pelletized. It has been reported that the sludge in a UASB reactor stratifies according to the sludge particle size: from large aggregates to fine suspended sludge particles (Andrews *et al.*, 1988). Larger and dense particles tend to be granular and occur mostly in the lower region of the sludge bed. Next up are the sludge flocs which are less dense than the granular ones. Lettinga *et al.* (1980) reported sludge volume indices of 10-20 and 20-40 ml g<sup>-1</sup> for granular and flocculant sludge. The same authors also found that granular sludge was superior to flocculant sludge in activity and settleability. Furthermore, two types of granules can form: granules comprising predominantly filamentous-type or rod-type microorganisms. The causes for the difference are not well understood (Andrews *et al.*, 1988).

Sludge granulation in the UASB reactor may be seen in three phases (Barford, 1988; Wu *et al.*, 1985):

- **First Phase.** This phase is the start-up period. The reactor is started with seed sludge. The organic loading rate at this time is low. The sludge bed expands upwards as gas production picks up. Filamentous organisms tend to grow, and hence sludge settleability deteriorates.
- **Second Phase.** As loading increases, poorly-settled sludge flocs become washed out the system; denser sludge particles start to compact at the bottom of the reactor and grow larger giving rise to the first new granules.
- **Third Phase.** As the loading is further raised to near the optimum, sludge granulation proceeds rapidly. Once the sludge is in the granular form, sludge washout reduces to a minimum; hence there is a positive net sludge growth. The reactor is then able to operate stably at high volumetric loading regimes.

Major factors affecting sludge granulation include seed sludge characteristics, the amount of seed sludge used at start-up, wastewater characteristics, and operational parameters and management (Lettinga *et al.*, 1985). These factors are each elaborated in the following sub-sections.

### 1) The Effect of Seed Sludge Characteristics

The choice of seed sludge is of great importance, especially in relation to start-up. Commonly, digested sewage sludges are used as seed materials. Lettinga *et al.* (1985) recommended the use of thick types of sludge with dry solids (DS) concentrations of 60 g DS liter<sup>-1</sup>. Thinner types of sludge (less than 40 g DS liter<sup>-1</sup>) are higher in methanogenic activity than the thicker types, but thicker sludges display better settleability than the thinner. In spite of its lower methanogenic activity, the thicker digested sludge will be retained more easily in the reactor allowing the development of more granules. When a thinner seed sludge is used, it takes relatively little time for the sludge bed to expand upward and throughout the entire reactor height. This excessive expansion implies that the sludge retention time is greatly

shortened and hence there may not be enough time for the development of well-settled bacterial aggregates. In this case, bacterial granulation can only take place via attachment to inert well-settleable support particles and to the reactor wall.

As far as speeding up the granulation process is concerned, two methods are mentioned by Lettinga *et al.* (1985). First, enhanced granulation may be realized by supplying to the reactor contents inert granular carrier material such as hydro-anthracite or gravel. This first method bases on bacterial attachment to inert well-settleable particles as explained above. Second, granulation will proceed more readily if a small amount of crushed granular sludge is added to the seed sludge. The added crushed granular sludge consists of small active nuclei which, if properly managed, will grow into larger granules and segregate from the more flocculant sludge.

## **2) The Effect of Amount of Seed Sludge**

The amount of seed sludge that is needed for a good start-up is not arbitrary. Normally, one would judge that, the more seed sludge one uses, the faster the start-up and better subsequent reactor performance will be. However, it has been shown that excess seed sludge leads to an unnecessarily expanded sludge bed which will tend be washed out the system. Conversely, too little seed sludge will cause the start-up to take very long. Existing experiences suggest a mixed liquor concentration of 12–15 g VSS liter<sup>-1</sup> if thick digested sewage sludge is used. For the case of a thin digested sewage sludge (less than 40 g TS liter<sup>-1</sup>), a 6 g VSS liter<sup>-1</sup> mixed liquor concentration should be maintained (Lettinga *et al.*, 1985).

## **3) The effect of Wastewater Characteristics**

The ease with which granulation occurs is highly waste-dependent. Barford (1988), Lettinga *et al.* (1980, 1985), and Wu *et al.* (1985) made the following important points:

- Granulation occurs most readily in wastes rich in soluble carbohydrate, such as sugar beet and potato processing effluents. In wastes like render waste, distillery waste, and corn starch waste, granulation proceeds at relatively slow pace.
- Granulation is a result of bacterial growth; therefore, all nutritional requirements (i.e., N, P, S, and trace elements) must be present in sufficient concentrations; otherwise, they must be supplemented to the wastewater. Influent nitrogen levels must be in the quantity of 30:1 to 55:1 in terms of COD:N ratio. When there is enough buffering capacity in the reactor mixed liquor, a COD:N ratio of 200:1 is acceptable. (In anaerobic digestion, buffering capacity is provided by ammonium hydroxide, which results from the breakdown of proteinaceous matters in the feed substrate.) The phosphorus requirement is commonly assumed to be one-fifth of the nitrogen requirement. As a general rule, the minimum concentrations of N, P, and S may be estimated based on the bacterial composition and growth yield factor. As to trace elements, the addition of Ni, Co, Mo, and  $ZnSO_4$  has led to positive results.
- The presence of low levels of  $Ca^{2+}$  (less than  $200\text{ mg liter}^{-1}$ ) has been found to exert a positive influence on granule formation. The probable reason is that  $Ca^{2+}$  may serve as a cementing agent providing mechanical strength for the flocs. However, when granular sludge is employed to treat a wastewater rich in  $Ca^{2+}$ ,  $CaCO_3$  scaling of the granules occurs. In addition, excess  $Ca^{2+}$  may raise the density of the sludge bed such that the contact between the incoming wastewater and the sludge bed becomes hindered. High concentrations of  $NH_4^+$  have also been found to be inhibitory to granulation.

#### 4) Operational Parameters

Operational parameters are vital in contributing to granulation; the following guidelines should be adhered to (Lettinga *et al.*, 1985; Wu *et al.*, 1985):

- pH should be kept between 6.5 and 7.8.
- For mesophilic digestion, the reactor operating temperature should be maintained in the optimal range of  $35^\circ\text{C}$  to  $40^\circ\text{C}$ .
- The control of sludge loading plays an important role in the speed at which granulation proceeds. According to the research done by Wu *et al.* (1985),

granules began to appear at a sludge loading of  $0.3 \text{ g COD g}^{-1} \text{ VSS day}^{-1}$  for brewery wastewater. The same finding was confirmed by other authors treating distillery and citric wastes. Moreover, Wu *et al.* (1985) suggested that after start-up the sludge loading rate be raised to over  $0.6 \text{ g COD g}^{-1} \text{ VSS day}^{-1}$  so as to speed up granulation.

- Hydraulic loading is important in that it can be used to impose a selection pressure on the system. Higher hydraulic loading rates have been proven to be beneficial to granulation. At higher hydraulic loading rates, light and bulking sludge tends to migrate upwards whereas heavy sludge tends to collect at the bottom of the reactor. Since the nutrients (the incoming wastewater) are introduced from the base of the reactor, the heavy sludge is allowed to get its food first and granulation takes place more quickly than when no selection pressure is imposed. Hydraulic loading rates of  $0.25$  to  $0.4 \text{ m}^3 \text{ m}^{-2} \text{ hr}^{-1}$  have been suggested. Where a wastewater is high in strength (higher than  $5 \text{ g COD liter}^{-1}$ ), pulse feeding is recommended (Wu *et al.*, 1985; Lettinga *et al.*, 1985).
- Granulation proceeds best when the feed wastewater strength is low:  $1.5$  to  $4 \text{ g COD liter}^{-1}$  is recommended. When the wastewater strength is low, continuous feeding should be employed; if the wastewater strength is high (over  $5 \text{ g COD liter}^{-1}$ ), pulse feeding or effluent recycle will be necessary. The purpose of using a dilute wastewater is to apply a high selection pressure on the system, i.e. relatively high hydraulic loading. The selection pressure promotes the washout of finely dispersed matter from the seed sludge and newly formed dispersed bacterial matter— while allowing the heavier sludge to remain in the reactor. The presence of high concentrations of inert and biodegradable suspended matter discourages (and in some cases inhibits) granulation.

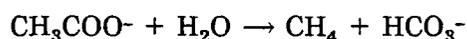
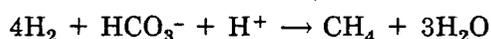
#### D. Sulfate Toxicity

Sulfurous compounds are commonly found in many industrial wastewaters (Sarner *et al.*, 1988; Lettinga *et al.*, 1985). When sulfurous organic compounds are biologically oxidized, they give rise to sulfate. Sulfate can be reduced to hydrogen sulfide (and elemental sulfur and/or organic sulfurous compounds, to a lesser extent) anaerobically by sulfate-reducing bacteria (SRB), which utilize sulfate as an electron acceptor. The electron donors are hydrogen and acetate, which are the main substrate for methanation. SRB have a greater affinity for hydrogen and acetate because sulfate

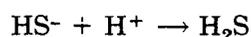
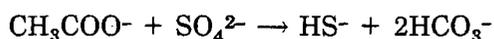
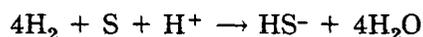
reduction is thermodynamically favored over the reduction of bicarbonate (Isa *et al.*, 1986).

The competition between methane production and sulfate reduction is shown below:

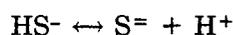
*Methane Production:*



*Sulfate Reduction:*



Sulfate itself does not pose the worst problem in anaerobic digestion. Lettinga *et al.* (1985) stated that sulfate levels below 5 g  $\text{SO}_4^{=}$  liter<sup>-1</sup> do not cause significant inhibition. Usually, the concern is focused on the main by-product of sulfate reduction,  $\text{H}_2\text{S}$ , and its dissociation species,  $\text{HS}^-$  and  $\text{S}^{=}$ , as shown below.



$\text{H}_2\text{S}$  is most toxic, followed by  $\text{HS}^-$ , which is in turn more toxic than  $\text{S}^{=}$  (Sarner *et al.*, 1988). It has been shown that, as long as the COD/ $\text{SO}_4^{=}$  ratio is above 10 g g<sup>-1</sup>,  $\text{H}_2\text{S}$  inhibition is rather insignificant. The reason is that  $\text{H}_2\text{S}$  is naturally removed from an anaerobic reactor through the so-called gas stripping process. As  $\text{H}_2\text{S}$  is produced in the reactor mixed liquor, it is mixed with the rest of the biogas and leaves the system. This built-in ability of removing  $\text{H}_2\text{S}$  is significantly reduced as the COD/ $\text{SO}_4^{=}$  ratio falls below 10 g g<sup>-1</sup>.

There are several ways through which  $H_2S$  toxicity can be controlled (Sarner *et al.*, 1988):

- Dilution of the wastewater helps alleviate the toxic effects of  $H_2S$ .
- Removal of  $H_2S$  through precipitation with the addition of metal ions such as  $BaCl_2$  (Lo *et al.*, 1989).
- Adjustment of pH to the range of 7.5-8, where most  $H_2S$  is in the less toxic form,  $HS^-$ . In practice,  $S^{=}$  may be disregarded because it exists in negligible levels for a pH below 8.
- External  $H_2S$  stripping (Lettinga *et al.*, 1985).

### III. MATERIALS AND METHODS

#### A. Feed Substrate

The feed was prepared from baker's yeast wastewater obtained from a local bakery yeast grower. Raw baker's yeast wastewater was diluted down to the desired COD concentrations ranging from 8 to 60 g liter<sup>-1</sup>. The characteristics of the raw wastewater are summarized in Table 3.1. Worth noting is the low BOD<sub>5</sub>-to-COD ratio (1:3 to 1:6), which implies that a good percentage of the wastewater consists of hard-to-degrade organics.

The concentration of nitrogen required to support biological growth is estimated as 30:1 to 55:1 in terms of COD:N ratio. Using a COD:N ratio of 45 and a raw wastewater concentration of 80,000 mg liter<sup>-1</sup>, the required nitrogen is calculated to be 1780 mg liter<sup>-1</sup>; then 355 mg liter<sup>-1</sup> of phosphorus is needed. As it was, the raw wastewater used seemed to contain adequate nitrogen (1,610-2,060 mg liter<sup>-1</sup>) but was lacking in phosphorus (only 108 mg liter<sup>-1</sup> of PO<sub>4</sub><sup>3+</sup>). As a nutrient supplement, NH<sub>4</sub>Cl and K<sub>2</sub>HPO<sub>4</sub> were added to the feed at proportions of 0.50 and 0.24 g liter<sup>-1</sup> respectively. The supplemented nitrogen (170 mg liter<sup>-1</sup> as NH<sub>4</sub><sup>+</sup>, by weight percentage) ensured that there was plenty of nitrogen. As for phosphorus, about 130 mg liter<sup>-1</sup> of phosphate was supplemented to the feed substrate. Because dilution of raw wastewater was always the case, nitrogen and phosphorus requirements would work out to be less than the numbers calculated above for full-strength wastewater. Hence the amount of phosphorus added was considered adequate.

Also the feed was pH-adjusted<sup>2</sup> to about 7 at the time of preparation, and it was stored at 4°C in a walk-in cooler. Whenever needed, a portion of the stored feed was transferred into the feed tank which in turn was kept in a refrigerator maintained

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<sup>2</sup> pH adjustment was done by adding concentrated NaOH solution to the feed which was thoroughly stirred and whose pH monitored by a pH meter.

between 2°C and 4°C. No batch of feed was ever kept for longer than 2 weeks. During the two-week period, it was found that the various characteristics of the feed (such as TSS, pH, and COD) did not change to any significant extent.

**TABLE 3.1:** Raw baker's yeast wastewater characteristics\*

5-day BOD.....	18,340-27,200
COD.....	70,000-100,000†
BOD-to-COD ratio.....	1:3 to 1:6
Total Suspended Solids.....	3,800-7,300
Total Kheldahl Nitrogen.....	1,610-2,060
Phosphate.....	108
Sulfate.....	1,700-3,200
pH.....	4.0-5.0

\* All, except pH, expressed in mg liter<sup>-1</sup>.

† 80,000 mg liter<sup>-1</sup> is the norm.

## B. Reactors

Both reactors were of UASB reactor design except that reactor B incorporated a fixed-film system to increase surface area within the reactor for bacteria to adhere onto. The UASB reactor configuration is depicted in Figure 3.1. The reactor was a cylinder made of acrylic plastic with an inner diameter of 11.5 cm and a total length of 168 cm. The working volume was 16.0 liters. Eleven sampling ports were installed along the length of each reactor starting from near the bottom. The spacing between two neighboring sampling ports was such that it widened towards the top of the reactor.

A feature in the reactor design that distinguishes UASB reactors from others is the conical three-phase separator located near the top of the reactor as indicated in Figure 3.1. The separator separates and collects the biogas produced from the wastewater. The biogas goes through a water column (which regulates the liquid level

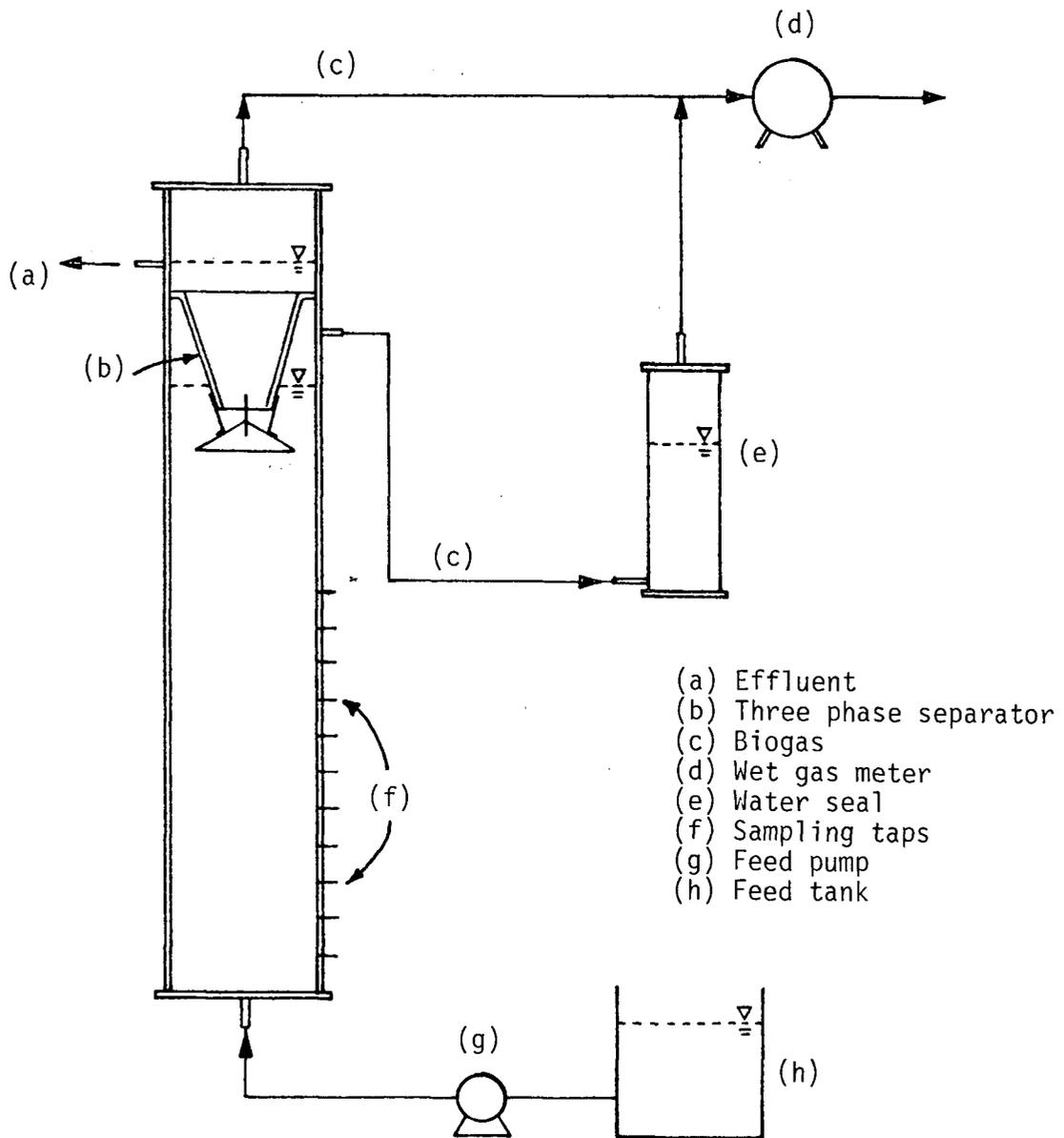


Figure 3.1: Configuration of UASB reactor

around the separator) and passes through a wet gasmeter. Furthermore, the separator is constructed in such a fashion that its conical base faces upwards. As the cross section area increases, the velocity of the fluid flow drops and causes the sludge to be retained in the reactor. The effluent overflows from the top of reactor and is collected in a container.

In addition to its basic UASB design, reactor B was equipped with a matrix of 19 parallel ropes (0.4 cm in diameter and 88.9 cm in length) in its mid section, beginning at a height of 33.0 cm measured from the base of the reactor. The ropes were intended to increase the surface area on which bacteria could attach. With the ropes, reactor B's surface-to-volume ratio was boosted from 32.1 (same for both reactors) to  $45.4 \text{ m}^2 \text{ m}^{-3}$ , an increase of 41.4%.

A peristaltic pump was used to continuously draw the feed from the feed tank, pass it through a copper-alloy coil immersed in a warm-water bath to bring the feed temperature up to near that of the reactor mixed liquor, and then introduce it into the reactor from the bottom. The reactor mixed liquor was kept at  $35^\circ\text{C}$  via a series of heating pads wrapped around the body of the reactor which were feed-back controlled through a multi-channel temperature controller.

### **C. Seed Sludge**

Sludge from the effluents of other laboratory-scale anaerobic reactors treating baker's yeast wastewater was incubated at room temperature (about  $20^\circ\text{C}$ ) in a glass jar for over a month before being seeded into the reactors. During this period the sludge was fed regularly with raw baker's yeast wastewater. The aim of the incubation of the sludge was two-fold: (1) growing a large enough quantity of seed sludge for use and (2) developing active bacteria in the form of granules. At the time of seeding, the seed sludge was mostly fine particles with some flocs and granules.

Each reactor received 5.1 liters of this seed sludge at  $11.4 \text{ g liter}^{-1}$  VSS; hence the initial amount of sludge in each reactor was 58.3 g (i.e., 3.6 g for each liter of reactor volume) in the form of volatile suspended solids<sup>3</sup>.

#### D. Operation

The reactors were continuously fed and run at a fixed hydraulic retention time (HRT) of 7 days which was about 2.3 liters of feed pumped in everyday. The low loading rate was thought to minimize perturbation in the reactor; perturbation could lead to excessive sludge washout before the sludge had time to establish itself.

This first period of time constituted the reactor start-up which lasted 46 days. The reactors were first fed with a feed at  $8 \text{ g COD liter}^{-1}$  until they stabilized. During this time period, the organic loading rate was kept relatively low and constant (other operating parameters also kept constant) so that the seeded sludge would have time to accustom to the new reactor environment and so that observations thus obtained would represent the true start-up behavior of the reactors.

Then, during the step-up operation period, the feed COD concentration was increased stepwise to 18, 25, 30, 35, 40, 45, 50, 55, to  $58 \text{ g COD liter}^{-1}$ . It was the intention of the author to investigate the entire loading range covered to find such things as the operating setting in which the gas production was maximum and the setting in which the reactor became unrealistically inefficient. Moreover, the loading regime was planned with the hope that total system failure might be reached within the loading range imposed.

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<sup>3</sup> VSS is widely accepted as a measure of amount of biosludge.

## E. Analysis

The same set of chemical analyses were done on both the feed and effluent. The total solids (TS), volatile solids (VS), total suspended solids (TSS), volatile suspended solids (VSS), BOD<sub>5</sub>, and SO<sub>4</sub><sup>=</sup> were determined according to the standard methods (A.P.H.A., 1975). Both total and soluble COD were determined by the colorimetric method (Knechtel, 1978). Biogas composition and volatile fatty acids (VFA) were analyzed on a Hewlett Packard 5890A gas chromatograph. The volume of biogas was measured with a wet gasmeter; the measured volume was corrected to the standard temperature and pressure (STP) which are 0°C and one atmosphere. Total Kjeldahl nitrogen (TKN) and ammonia nitrogen (NH<sub>3</sub>-N) were determined using a block digester and a Technicon Auto Analyzer II respectively (Schumann *et al.*, 1983).

BOD<sub>5</sub> and SO<sub>4</sub><sup>=</sup> tests were done only as a check; no results have been shown in this thesis.

## IV. START-UP

The start-up period was meant to help the seeded sludge to multiply and, at the same time, acclimate to the new reactor environment. At the beginning of the start-up, each reactor was seeded with 58.3 g of sludge which had previously been incubated for the purpose. The feed strength was maintained at 8 g COD liter<sup>-1</sup> at an average HRT of 7 days. The relatively low feed strength was intended to help minimize loading shock and buoyancy effects caused by gas bubbles in the reactor mixed liquor, and thus establish an active and, preferably, granular sludge as early as possible.

The characteristics of the feed for the start-up period are summarized in Table 4.1.

**TABLE 4.1: Start-up feed Characteristics**

pH.....	7.0
VS (g liter <sup>-1</sup> ).....	6.91
VSS (g liter <sup>-1</sup> ).....	1.52
COD (g COD liter <sup>-1</sup> )	
Total.....	8.11
Soluble.....	7.03
BOD (g BOD liter <sup>-1</sup> ).....	2.51
BOD-COD ratio.....	1:3
VFA (mg liter <sup>-1</sup> )	
Acetic.....	301
Propionic.....	43
Isobutyric.....	7
Butyric.....	29

### A. Results and Discussions

Existing literature on the anaerobic treatment of baker's yeast wastewater utilizing the UASB reactor is very scarce, rendering comparison between results found in this study and those from the literature extremely difficult. Therefore, comparison has mostly been done between the two reactors— mainly to identify any effects of the ropes in reactor B.

## 1) Biogas Methane Composition

The biogas methane composition can serve as an indicator of the stabilization of the fermentation process (Figure 4.1). It is clear that the methane percentage<sup>4</sup> in biogas increased essentially monotonically from 14.5% and 24.0% on day 1, A and B respectively, to 59.0% and 55.1% on the 46th day. Noteworthy is the presence of a small dip for both reactors around day 10; after the dip, the methane content climbed rather rapidly until day 18. Thereafter, the methane content appeared to have come to a plateau with some minor depression near day 30 for reactor B. Information about reactor A at this point was not available because a gas leak was detected between day 18 and 38. Hence, the missing points were thought best represented through linear interpolation.

Steady state during the start-up has been defined as the state of the reactor system (the microorganisms and reactor environment) in which the system parameters (methane production, VFA levels, etc) did not vary by more than 10% over a time span of three or four days. Basing merely on Figure 4.1, reactor B virtually achieved steady state after day 17, but reactor A did not reach steady state until after day 43. Basing calculations on days 43 to 46, the corresponding mean methane compositions achieved were 59.1% and 54.8% for reactors A and B respectively. A summary of all steady-state data is found in Table 4.2.

On the basis of the methane compositions between days 43 and 46 alone, it appeared that reactor B did not perform better than reactor A. However reactor B's volumetric production rate was significantly higher than that of reactor A ( $0.28 \pm 0.01$  versus  $0.23 \pm 0.02$  liter  $\text{CH}_4$  liter<sup>-1</sup> day<sup>-1</sup>). The implication here is that if reactor B were to achieve the same methane yield as reactor A, reactor B would have to, and in fact did, sustain a proportionally higher COD utilization. In addition, as hinted at

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<sup>4</sup> Throughout the thesis, biogas methane composition was always expressed as percentage on a volume-to-volume basis.

# BIOGAS METHANE COMPOSITION

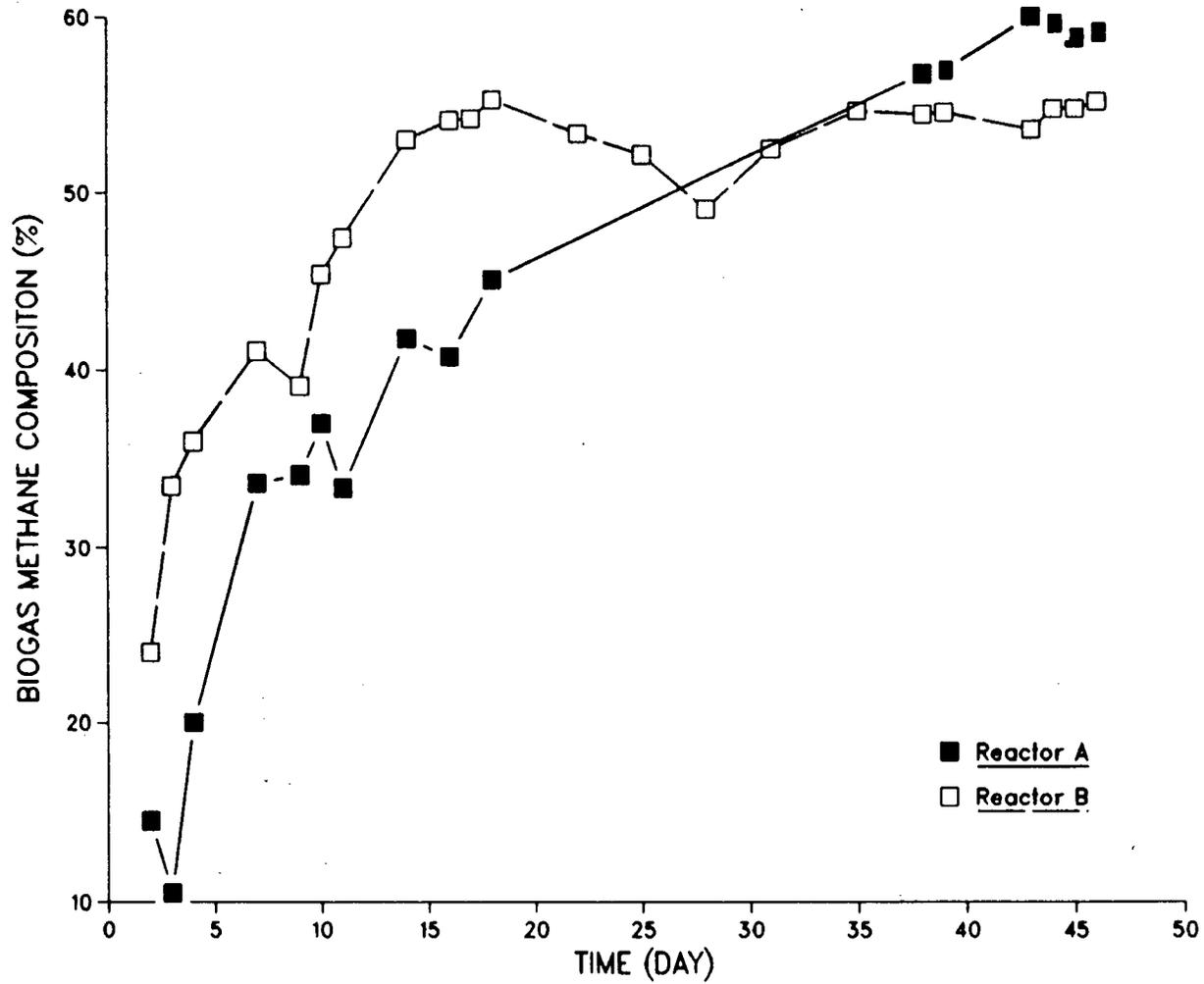


FIGURE 4.1: Biogas Methane Composition versus Time

earlier, by day 18 reactor B had already attained a methane composition of 55.3%, which was essentially reactor B's steady-state value (54.8%). With reactor A, however, a methane composition of only 45.0% was achieved by day 18. In short, according to methane composition data alone, it took far less time for reactor B to arrive at steady state. Thus the merit of the fixed medium in the form of ropes in reactor B could not be dismissed. However, it should be noted that COD reduction and the related parameters such as methane yield are more important from an operator's standpoint.

**TABLE 4.2:** Average steady-state data summary of start-up period

	A	B
HRT (days).....	7.35	6.42
Waste strength (g COD liter <sup>-1</sup> ).....	8.11	8.11
Loading Rate (g COD liter <sup>-1</sup> day <sup>-1</sup> ).....	1.11 ± 0.05	1.27 ± 0.03
Methane Content (% v/v).....	59.1 ± 0.4	54.8 ± 0.2
Methane Production Rate (liter CH <sub>4</sub> liter <sup>-1</sup> day <sup>-1</sup> ).....	0.23 ± 0.02	0.28 ± 0.01
Methane yield (liter CH <sub>4</sub> g <sup>-1</sup> COD destroyed).....	0.34 ± 0.03	0.33 ± 0.01
Effluent Characteristics		
pH.....	7.82 ± 0.06	7.81 ± 0.05
VS (g liter <sup>-1</sup> ).....	3.1 ± 0.1	2.8 ± 0.1
VSS (g liter <sup>-1</sup> ).....	0.3 ± 0.1	0.2 ± 0.01
COD (g COD liter <sup>-1</sup> )		
Total.....	3.08 ± 0.16	2.67 ± 0.07
Soluble.....	2.95 ± 0.05	2.68 ± 0.09
BOD (g liter <sup>-1</sup> ).....	0.99	0.75
VFA (mg liter <sup>-1</sup> )		
Acetic.....	61 ± 7	44 ± 8
Propionic.....	48 ± 34	10 ± 6
Isobutyric.....	0	0
Butyric.....	0	0
COD Reduction (%)		
Total.....	62.1 ± 2.0	67.1 ± 0.9
Soluble.....	58.0 ± 0.8	61.9 ± 1.2
BOD Reduction (%).....	60.6	70.1

Yan *et al.* (1989a) conducted a start-up study of a UASB which treated cheese whey mesophilically using a 14.5-liter UASB reactor of rather similar configuration. The reactor was operated for 48 days, which were considered the start-up period, at organic loading rates of 0.91 and then 1.97 g COD liter<sup>-1</sup> day<sup>-1</sup>. The methane compositions achieved for the two organic loading rates were 56.5% and 48.2%. These numbers were comparable with those obtained in this thesis experiment: at similar organic loading rates of 1.11 and 1.27 g COD liter<sup>-1</sup> day<sup>-1</sup> for reactors A and B, the corresponding methane compositions of 59.1% and 54.8% have been attained.

## 2) Effluent COD

The establishment of the biomass in the reactors was reflected partly in the consistent lowering of effluent COD. The amount of biomass at a point in time can be estimated by such sludge sample tests as COD, VS, and VSS. Specific discussion pertaining to sludge present in the reactor is found in the session entitled "Reactor Profile Study". As bacteria utilize nutrients in the feed wastewater for maintenance energy and cell material synthesis, the organic strength of the feed decreases. But this decline in organic waste strength will only take place if the bacteria themselves do not get washed out of the reactor excessively and end up in the effluent stream. When excessive sludge washout occurs, even though COD removal is not insignificant, the resulting effluent COD may still be higher than that of the influent. The lack of good settling characteristics of the sludge is common during start-up and contributes to an overexpansion of the sludge bed and hence excessive sludge loss.

Both total and soluble effluent COD's showed the same pattern (Figures 4.2 and 4.3) for both reactors. Of special interest is that the effluent COD levels during the first few days were found to be higher than that of the feed. As time progressed, a transition was observed, after which the effluent total COD's decreased steadily to a

level of  $3000 \text{ mg liter}^{-1}$ . Similarly the percent COD reduction (Figures 4.4 and 4.5) pointed to the same conclusion. Its pattern is the opposite of that of Figures 4.2 and 4.3. Both total and soluble COD reductions increased consistently from around -20% to 60%; then they remained relatively constant. Based on the total-to-soluble-COD ratio and VSS (to be discussed in a later section), there was excessive sludge washout between days 10 and 15. The negative values also suggested that, at the beginning of the start-up phase, the 5.1 liters of seed sludge slurry probably contained a high concentration of colloidal and soluble COD, so that, when the reactor was filled up to its capacity, the overall COD concentration of the mixed liquor was substantially higher than that of the feed. Consequently, during the first few days, the effluent soluble and total COD's were higher than their influent counterparts. However, over time, as new feed was introduced, the initially high COD concentration was gradually "diluted", giving rise to a negative-to-positive turnover.

While both reactors display the same pattern in total and soluble COD, the distinct and consistent difference evidences reactor B's superior performance. It is unmistakable from Figures 4.2 and 4.3 that reactor B was consistently lower than reactor A in both total and soluble COD. Similarly, reactor B always achieved higher reduction both in total and soluble COD (Figures 4.4 and 4.5). As a whole, each pair of curves in Figures 4.2 through 4.5 follow each other very closely, looking almost parallel to each other.

During steady-state, the effluent total COD values for A and B were 3.1 and  $2.7 \text{ g COD liter}^{-1}$  while total COD reduction values were 62.1% and 67.1%. According to Table 4.2, reactor B's numbers are statistically different and better than those of reactor A. Not only so, the pathways by which the reactors arrived at those values strongly suggested that the ropes did have a positive effect on reactor B.

# EFFLUENT COD (TOTAL)

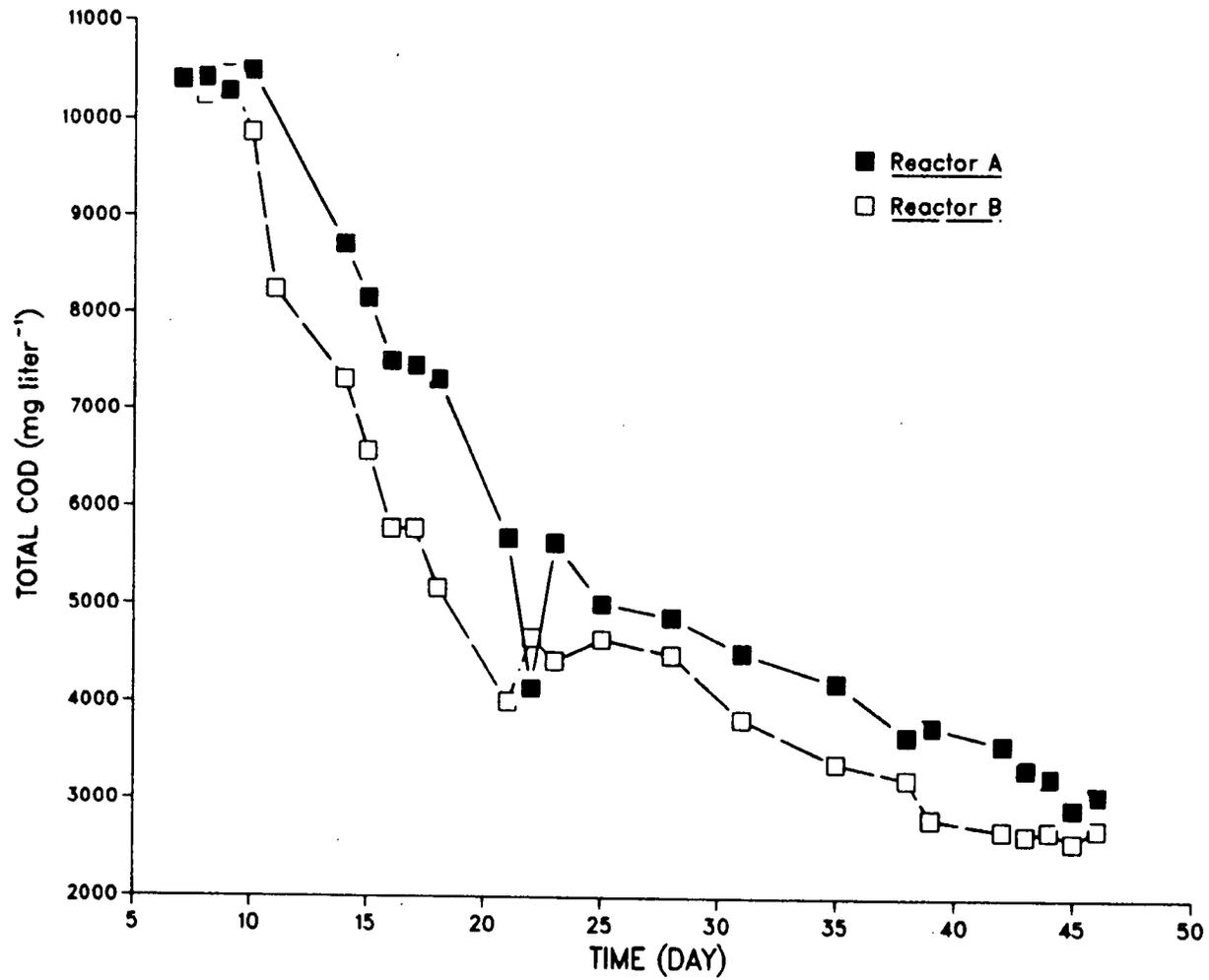


FIGURE 4.2: Total COD versus Time

# EFFLUENT COD (SOLUBLE)

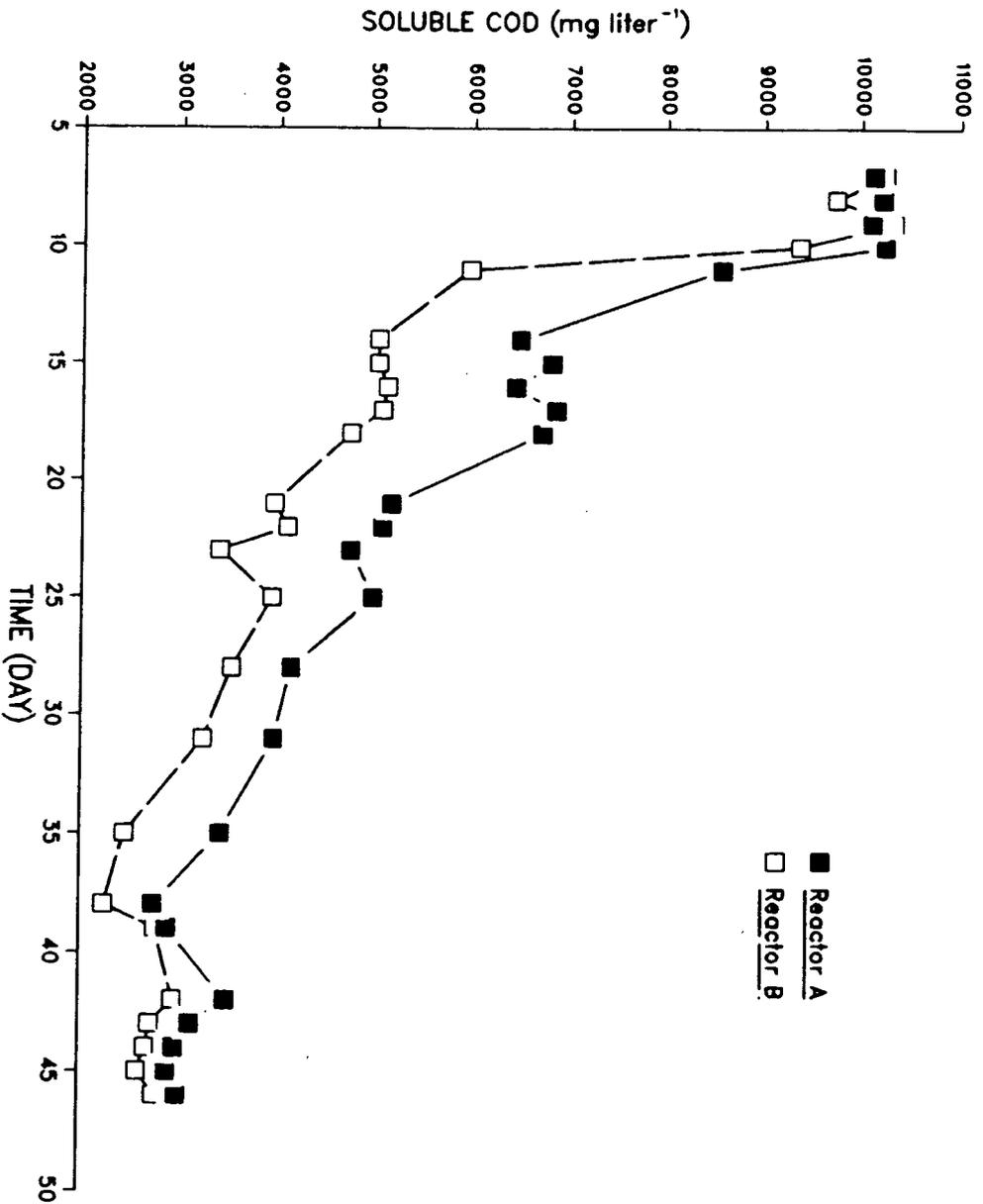


FIGURE 4.3: Soluble COD versus Time

# EFFLUENT COD REDUCTION (TOTAL)

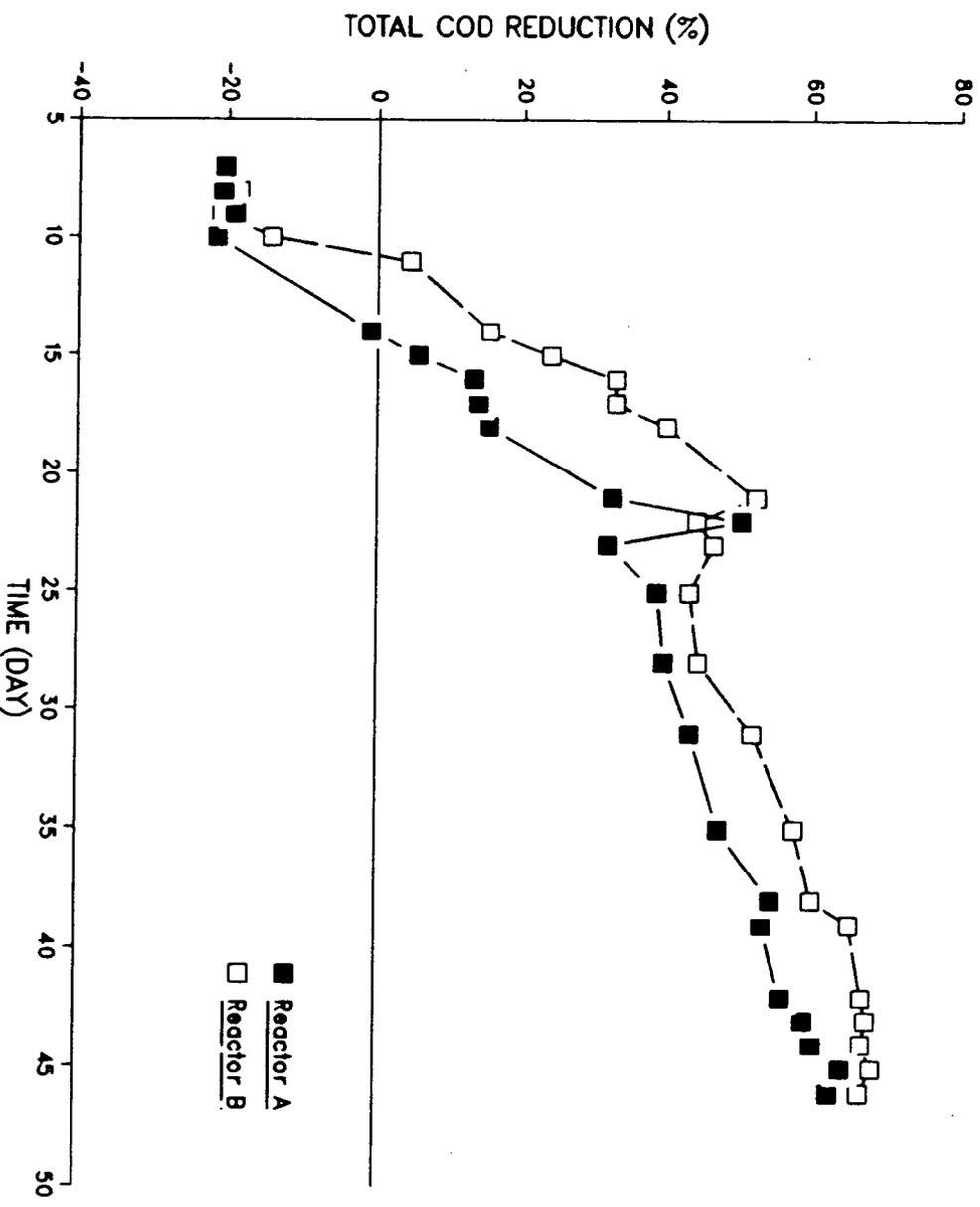


FIGURE 4.4: Total COD Reduction versus Time

# EFFLUENT COD REDUCTION (SOLUBLE)

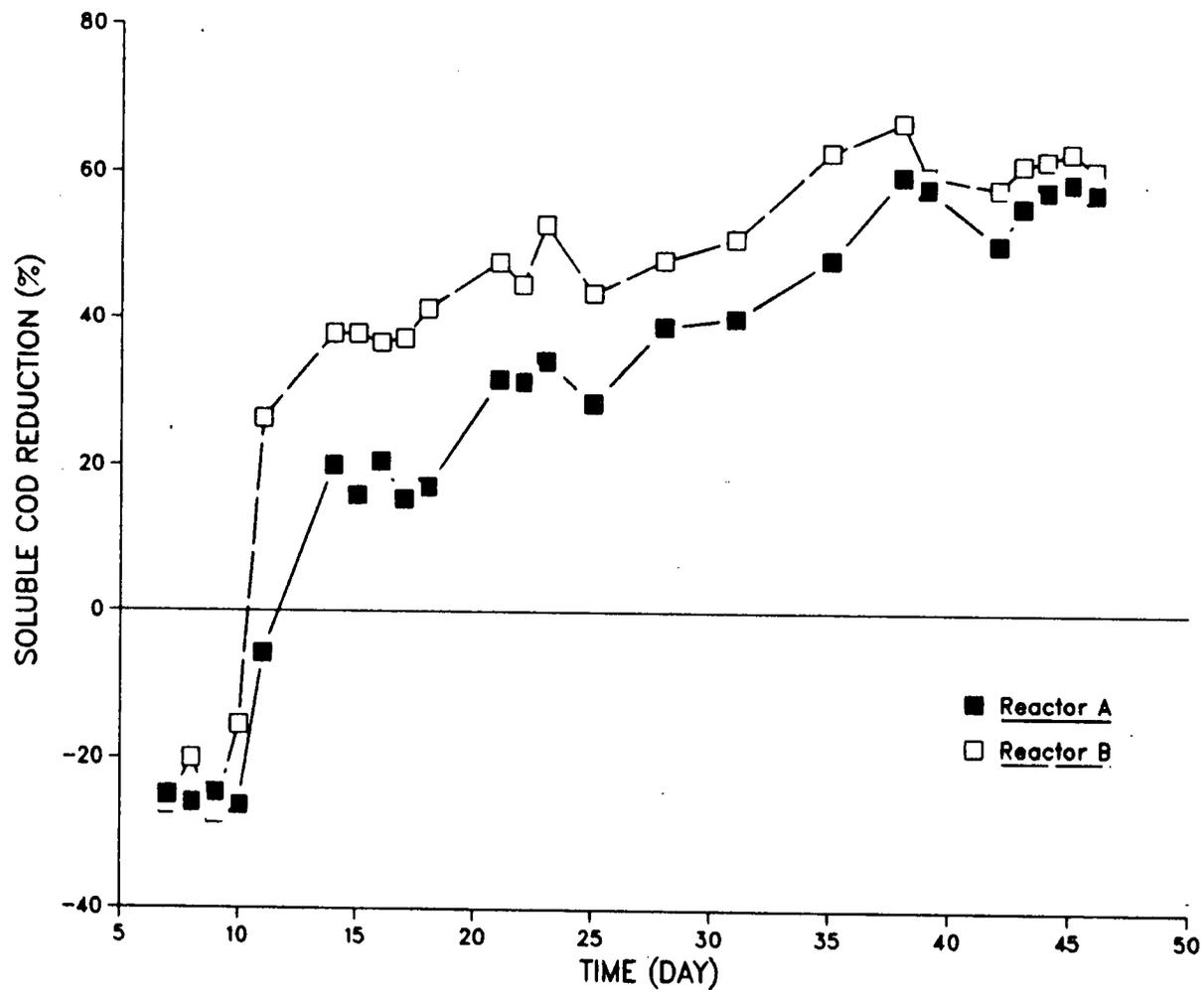


FIGURE 4.5: Soluble COD Reduction versus Time

### 3) Methane Yield

Figures 4.6 and 4.7 depict methane yield for the two reactors expressed in liter  $\text{CH}_4 \text{ g}^{-1} \text{ COD day}^{-1}$ , where COD is added or destroyed<sup>5</sup>. The per-COD-destroyed methane yield has been preferred and used for this discussion because it reflected the extent to which sludge was being washed out of the reactors.

Both reactors showed the same trend in methane yield, the only difference being in timing. The methane yield started off by going through a negative depression. Then recovery in the form of a negative-to-positive turnover was observed and was followed by stabilization (a plateau) which lasted for the remainder of the start-up period. As indicated already, from days 18 to 38, there was a gas leak in reactor A; therefore, Figures 4.6 and 4.7 employed linear interpolation for reactor A during this time interval.

The methane yield values for both reactors began slightly negative, -0.18 and -0.23 liter  $\text{CH}_4 \text{ g}^{-1} \text{ COD day}^{-1}$  for A and B respectively. Then the methane yield values continued to fall for both reactors. Reactor B hit its lowest of -0.60 liter  $\text{CH}_4 \text{ g}^{-1} \text{ COD day}^{-1}$  at day 10; reactor A, however, would not reach its lowest of -4.59 liter  $\text{CH}_4 \text{ g}^{-1} \text{ COD day}^{-1}$  until day 14. After the reactors had reached their lowest methane yield values, they each commenced to recover by going through a negative-to-positive turnover. Both the COD reduction (Figures 4.4 and 4.5) and methane yield (Figures 4.6 and 4.7) indicate that the turnovers occurred near days 15 and 10 for reactors A and B respectively. The first methane yield values immediately after the turnovers were 0.33 and 1.13 liter  $\text{CH}_4 \text{ g}^{-1} \text{ COD day}^{-1}$  for A and B respectively. Hereafter, the methane yield values underwent some decrease up to day 18 (down to 0.20 and 0.31 liter  $\text{CH}_4 \text{ g}^{-1} \text{ COD day}^{-1}$  for A and B); then they began to stabilize and eventually reached the steady-state with methane yield values relatively constant

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<sup>5</sup> COD destroyed means COD utilized.

# METHANE YIELD (COD ADDED)

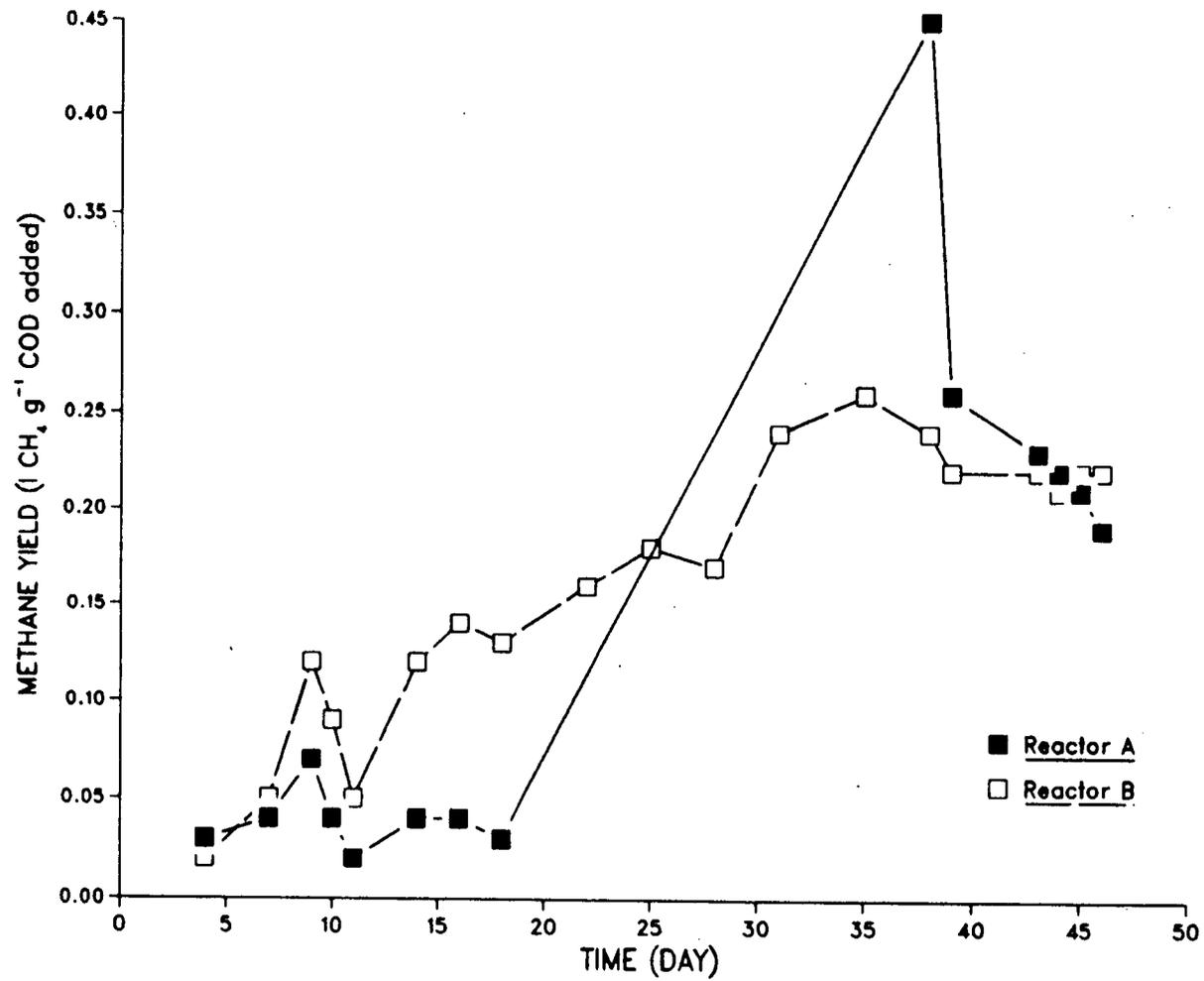


FIGURE 4.6: Methane Yield Based on COD Added versus Time

# METHANE YIELD (COD DESTROYED)

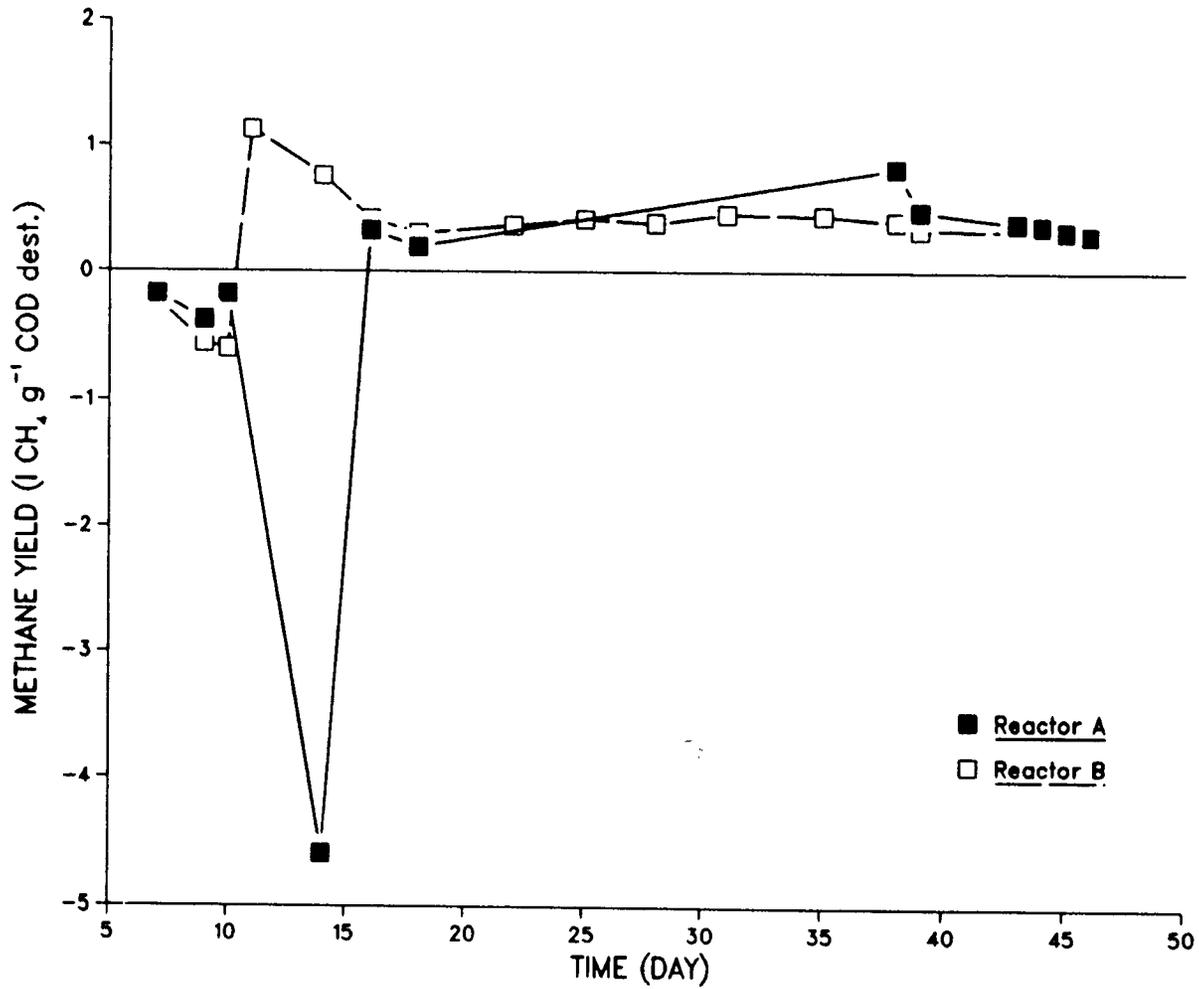


FIGURE 4.7: Methane Yield Based on COD Destroyed versus Time

ranging between 0.3 and 0.4 liter  $\text{CH}_4 \text{ g}^{-1} \text{ COD day}^{-1}$  (Table 4.2). Methane yield values of reactors A and B practically merged.

The negative methane yield values observed in the first 15 days were due to relatively large amounts of washed-out sludge in the effluent stream. Washed-out sludge contributed to the COD of the effluent. In effect, COD in the form of washed-out sludge was so high that it more than offset COD removed by bacteria, giving rise to negative COD reduction and hence negative methane yield.

The unusually large magnitudes of the peak methane yield values (positive and negative) had to do with the definition of methane yield itself. Methane yield is the quotient of  $\text{CH}_4$  produced and COD destroyed (or removed), the former being the numerator and the latter the denominator. Therefore, an unusually small negative value of COD destroyed would lead to a large negative value of methane yield (e.g., -4.59 liter  $\text{CH}_4 \text{ g}^{-1} \text{ COD day}^{-1}$  for reactor A). Similarly, a very small positive value of COD destroyed would produce a large positive value of methane yield (e.g., 1.13 liter  $\text{CH}_4 \text{ g}^{-1} \text{ COD day}^{-1}$  for reactor B).

Despite the fact that steady-state methane yield values for the two reactors were virtually the same, when comparing one reactor with the other, two remarks may be made. First, time needed for recovery from negative methane yield was shorter for reactor B than for reactor A— 10 days for reactor B and 15 days for reactor A. Second, whereas reactor B experienced a slight negative swing (-0.60 liter  $\text{CH}_4 \text{ g}^{-1} \text{ COD day}^{-1}$ ), reactor A went through a sharp valley (-4.59 liter  $\text{CH}_4 \text{ g}^{-1} \text{ COD day}^{-1}$ ). Moreover, after its negative-to-positive turnover, reactor B had achieved a positive peak of 1.13 liter  $\text{CH}_4 \text{ g}^{-1} \text{ COD day}^{-1}$  before starting to stabilize. These observations all point to the advantages that reactor B had been enjoying because of the fixed medium installed in it. In summary, with the ropes as fixed-medium to enhance attachment area for bacteria, reactor B displayed superior performance in the

form of shorter time needed for negative-to-positive turnover to occur, and lessening of variability in methane yield.

A methane yield range between 0.30 and 0.33 liter  $\text{CH}_4 \text{ g}^{-1} \text{ COD day}^{-1}$  was reported by Yan *et al.* (1989a) for the start-up of their UASB reactor which treated cheese whey at 35°C. Their organic loading rates of 0.91 and 1.97 g COD liter<sup>-1</sup> day<sup>-1</sup> were comparable to those applied to reactors A and B in this study (1.11 and 1.27 g COD liter<sup>-1</sup> day<sup>-1</sup>). It was interesting to note that the corresponding methane yield values in this study were 0.34 and 0.33 liter  $\text{CH}_4 \text{ g}^{-1} \text{ COD day}^{-1}$  for the two reactors. Whereas the reactor Yan *et al.* (1989a) operated was producing at 1.37 and 3.25 liter  $\text{CH}_4 \text{ liter}^{-1} \text{ day}^{-1}$ , reactors A and B produced only 0.23 and 0.28 liter  $\text{CH}_4 \text{ liter}^{-1} \text{ day}^{-1}$  (Table 4.2). Despite the contrast in volumetric methane production between reactors of the two parties, the methane yield numbers were very close to one another. In effect, this observation implies that the theoretical treatment efficiencies in terms of methane yield were the same for the two different sets of reactors utilizing two drastically different wastewaters. The author did not find literature which specifically dealt with baker's yeast wastewater treatment during start-up in UASB reactors which was based on comparable loading regimes.

#### 4) Volatile Suspended Solids

VSS information is useful in that it gives an indication of the concentration of sludge in the sample being examined. VSS, however, makes no distinction between living and dead bacteria. It is beyond the scope of this study to make such distinction.

Figure 4.8 depicts the effluent VSS over the entire start-up in which the influent VSS<sup>6</sup> stayed relatively constant at 1.5 g liter<sup>-1</sup>. The reactors started off with a VSS concentration in the neighborhood of 0.5 g liter<sup>-1</sup>. Next, these initially low VSS

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<sup>6</sup> Both VSS and VS concentrations (g liter<sup>-1</sup>) are based on the the assumption that the density of the sample is near that of water.

values began to rise and soon (between day 10 and 15) gave way to abrupt peaks of 2.9 and 3.1 g liter<sup>-1</sup> for reactors A and B respectively. After the peaks subsided on day 15, VSS levels essentially remained below 0.5 g liter<sup>-1</sup> until the close of the start-up period. The steady-state VSS values were marginally different: 0.3 and 0.2 g liter<sup>-1</sup>, A and B respectively.

The VSS behaviors of the two reactors were found to be similar. During the period of start-up, the seeded sludge was allowed to multiply as much as possible. As the sludge concentration increased in the reactor, higher flotation due to increasing gas production was responsible for the lifting and expansion of the sludge bed, and the associated excessive sludge washout especially between days 10 and 15. However, as the bacteria began to adapt to the reactor environment, the expanded sludge bed gradually became compacted leading to less and less sludge washout (Barford, 1988; Wu *et al.*, 1985).

The excessive sludge loss common in both reactors was also reflected in Figure 4.9. In the course of the first 10 days, VS<sup>7</sup> levels were consistently higher than that of the feed (6.9 g liter<sup>-1</sup>), implying abundant sludge washout, on which Figures 4.4 through 4.9 agreed. Behaving in a manner opposite to that of COD reduction, the VS graph goes from high to low, the slope of the graph gradually diminishing to about nil (i.e., flat) by day 40. This pattern makes good sense because the consistent lowering of VS implies a continuous growth of the bacterial population, its adaptation to the reactor environment, and its increasing fermentation activity. Finally, the establishment of a reasonably stable flat platform in VS signalled the completion of the start-up period. The steady-state VS concentrations were 3.1 and 2.8 g liter<sup>-1</sup>, A and B respectively.

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<sup>7</sup> VS is total volatile solids, which consists of two components: dissolved and suspended (i.e., VSS).

# VOLATILE SUSPENDED SOLIDS

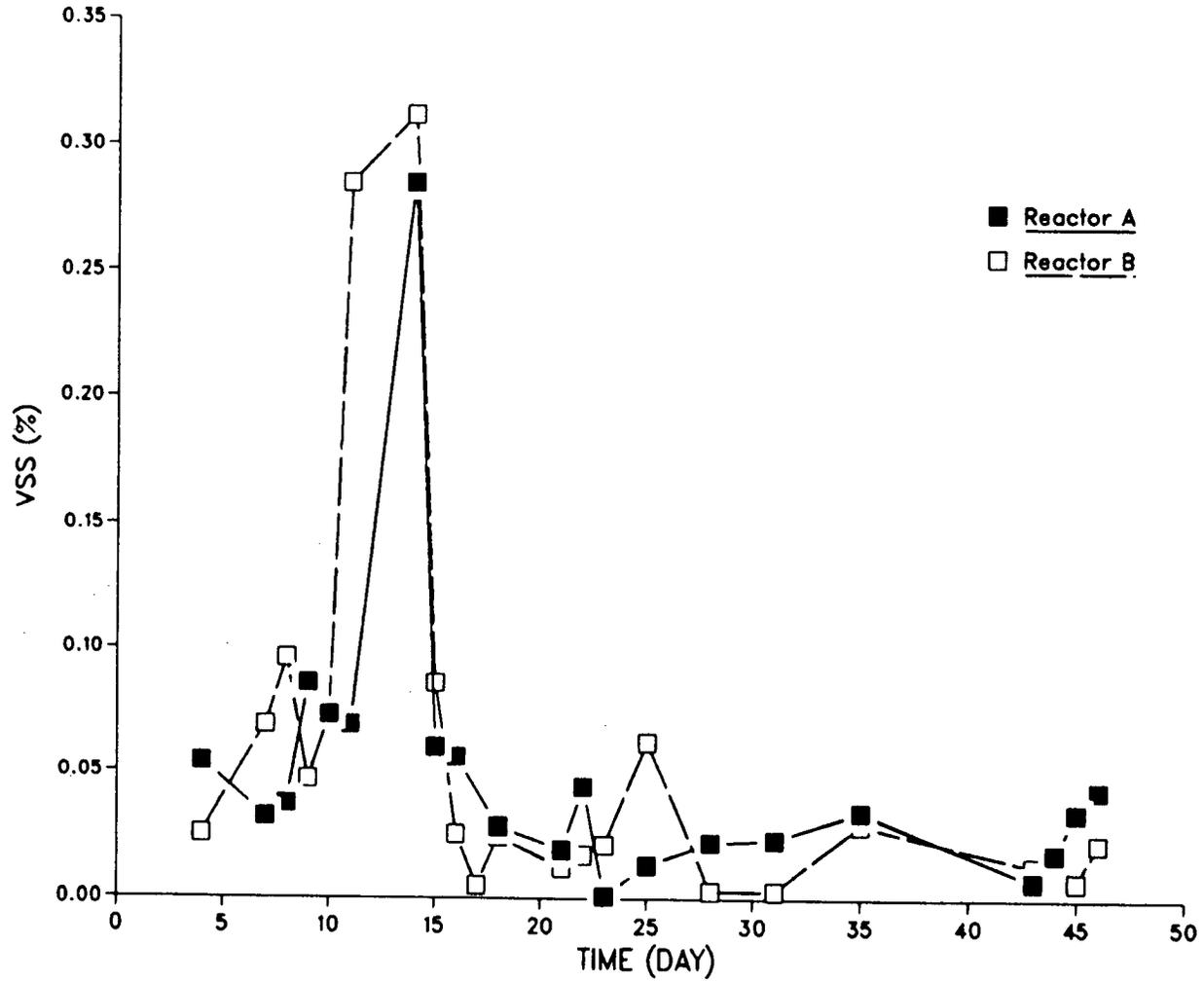


FIGURE 4.8: Volatile Suspended Solids versus Time

# VOLATILE SOLIDS

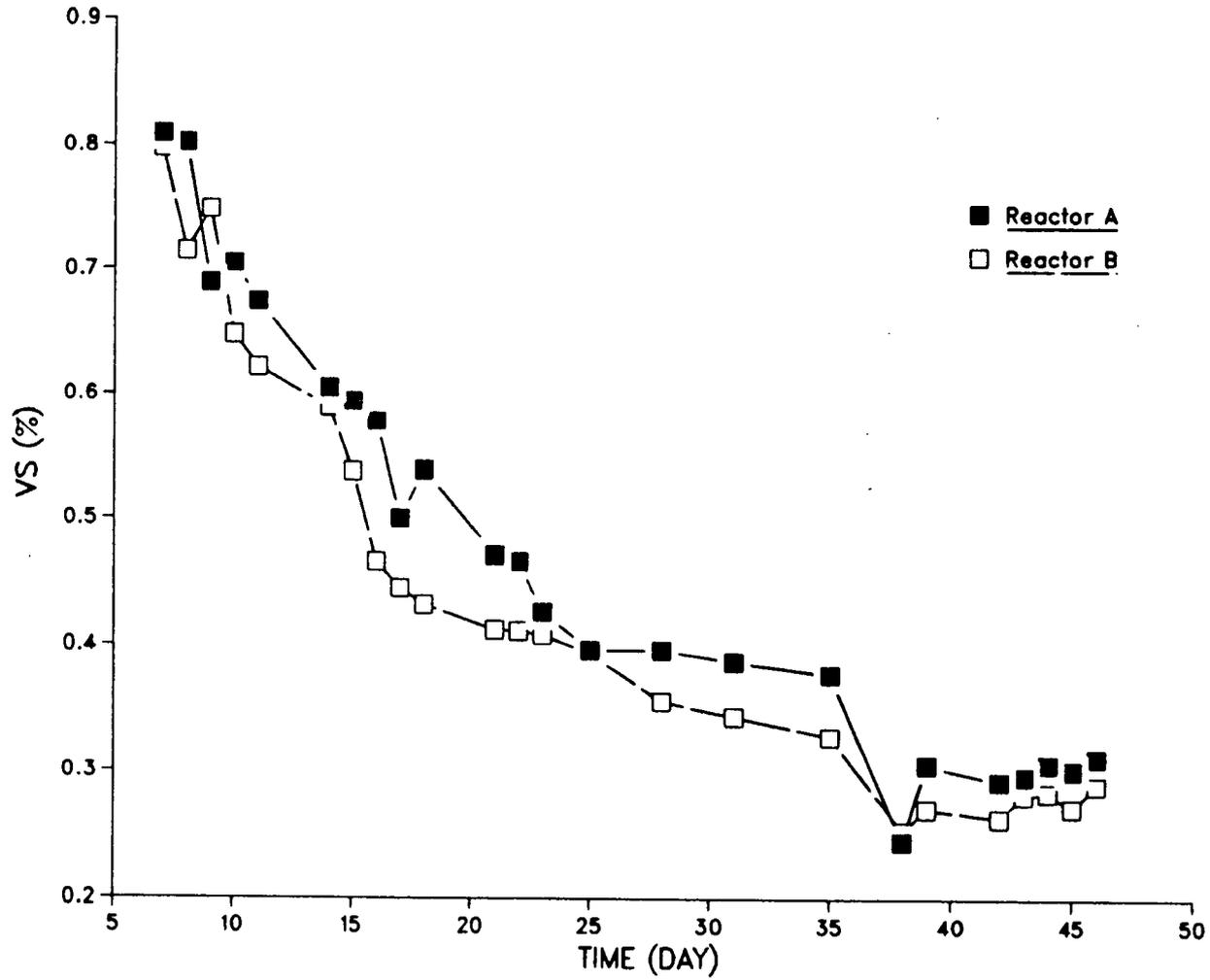


FIGURE 4.9: Volatile Solids versus Time

From the vantage point of effluent VSS and VS (Figures 4.8 and 4.9), there was no significant difference between the two reactors before and during the steady-state was established, even though undeniably better performance on reactor B's part was evident in effluent COD, effluent COD reduction, and methane yield (Figures 4.2 through 4.7).

### 5) Volatile Fatty Acids, pH, and P/A Ratio

Figures 4.10 and 4.11 indicate that both reactors began with very high effluent VFA concentrations in the form of acetic, propionic, iso-butyric, and butyric acids. However, as the biomass grew in concentration leading to increasing digestion activity, these VFA's were digested in accordingly greater proportions. Thus, the overall VFA behavior was the consistent lowering of all four VFA's throughout the first forty days. From day 42 onward, though, all effluent VFA levels from both reactors were found to converge to their respective steady-state concentrations. Except that isobutyric and butyric acids were zero in concentration for both reactors, the respective acetic and propionic acid concentrations for A and B were 61 and 48 mg liter<sup>-1</sup>, and 44 and 10 mg liter<sup>-1</sup>.

In addition, during the steady-state, the propionic acid concentration went lower than that of the acetic acid, and pH rose to between 7.5 and 8.0 (Figure 4.12). Less obvious is that reactor B's VFA levels reached stable conditions more quickly reactor A; that is, by day 35, reactor B's VFA levels were already below 200 mg liter<sup>-1</sup>, whereas the same would not happen for reactor A until after 40 days. This difference can be verified more easily from Figure 4.13 which is the time profile of propionic-to-acetic-acid (P/A) ratio. As mentioned in the literature review chapter, a P/A ratio of 1.4 or higher means that the anaerobic process is under stress. Here reactor B had its peak at day 22 whereas reactor A reached its peak at day 35.

## VOLATILE FATTY ACIDS (Reactor A)

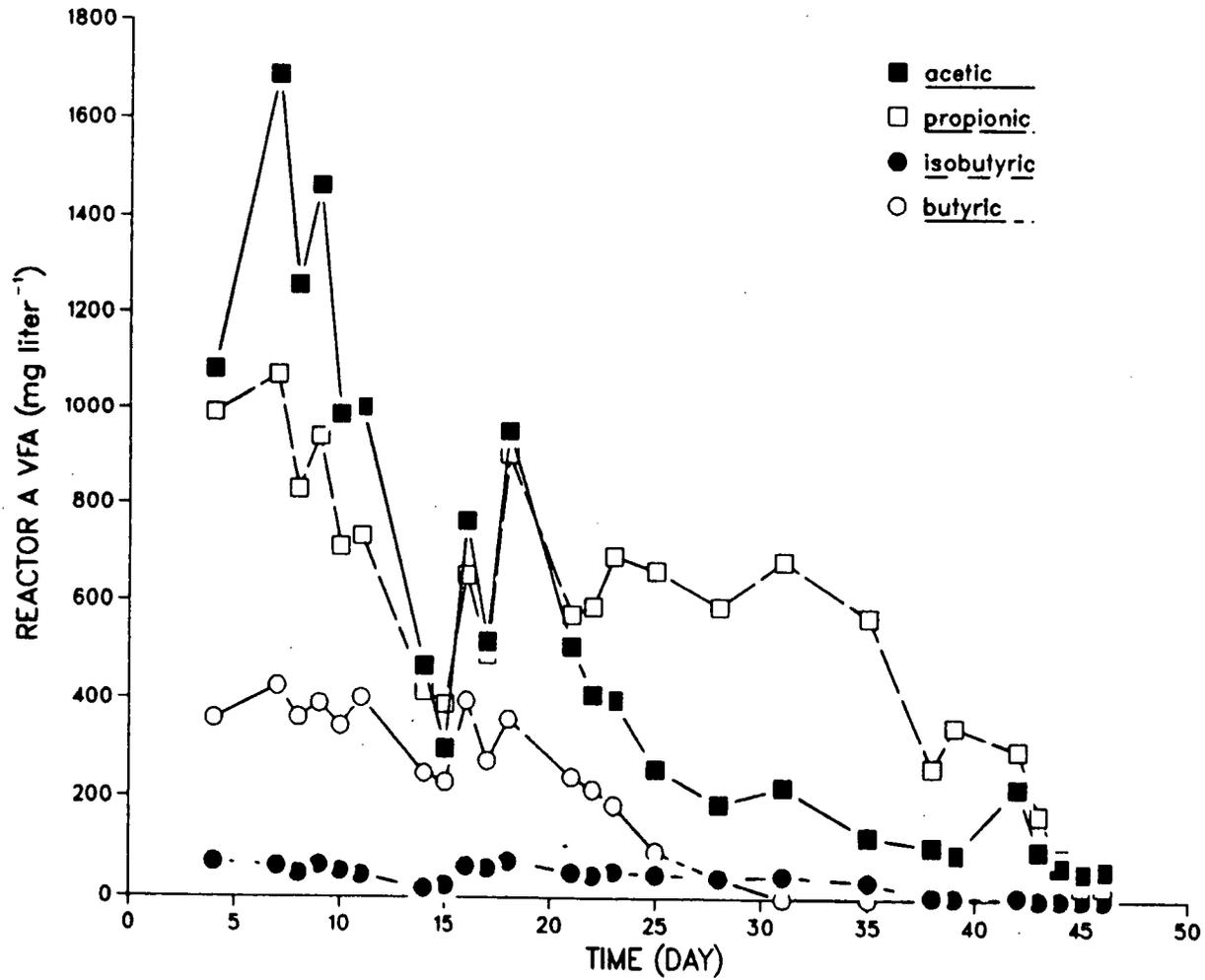


FIGURE 4.10: Volatile Fatty Acids versus Time for Reactor A

## VOLATILE FATTY ACIDS (Reactor B)

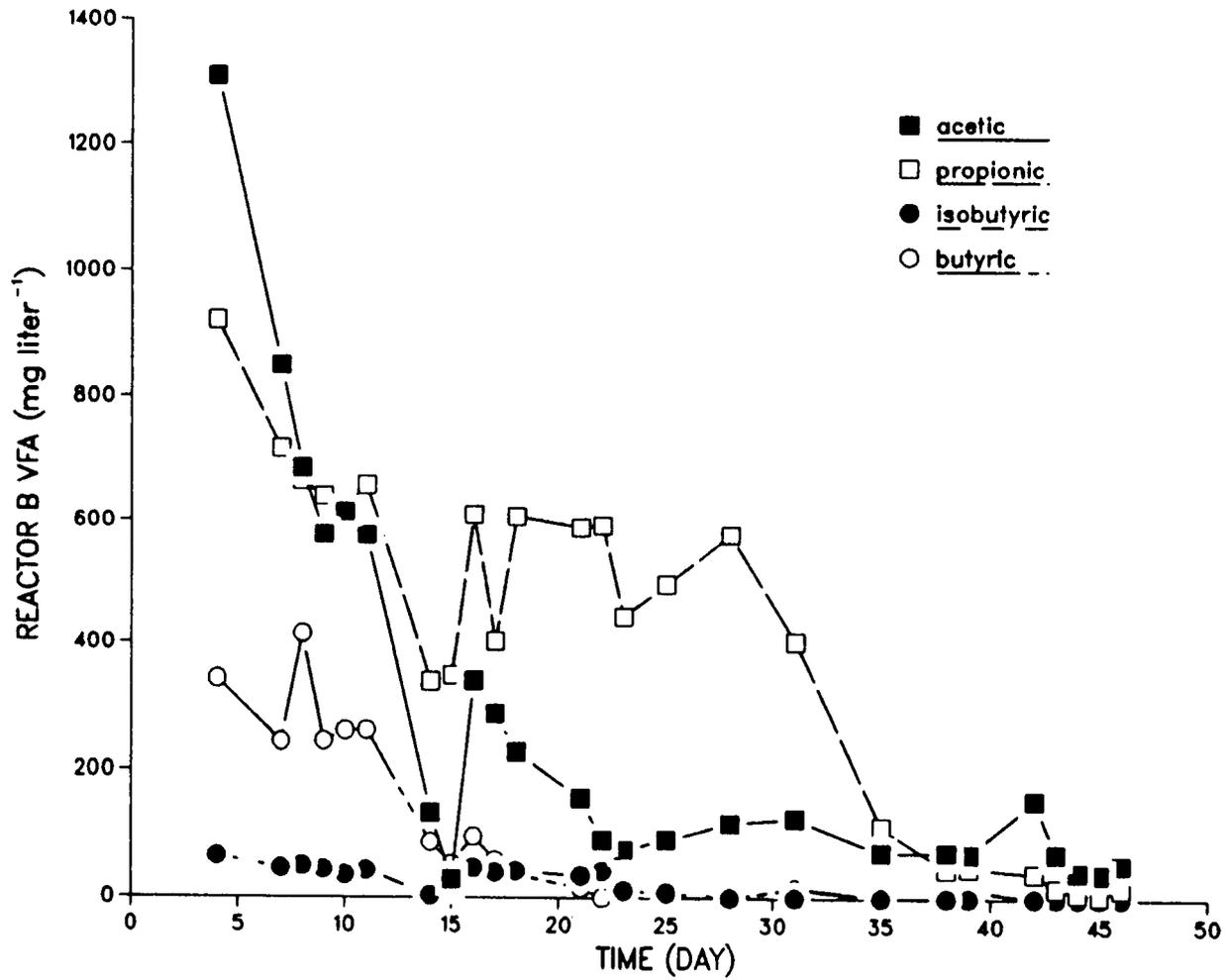


FIGURE 4.11: Volatile Fatty Acids versus Time for Reactor B

# EFFLUENT pH

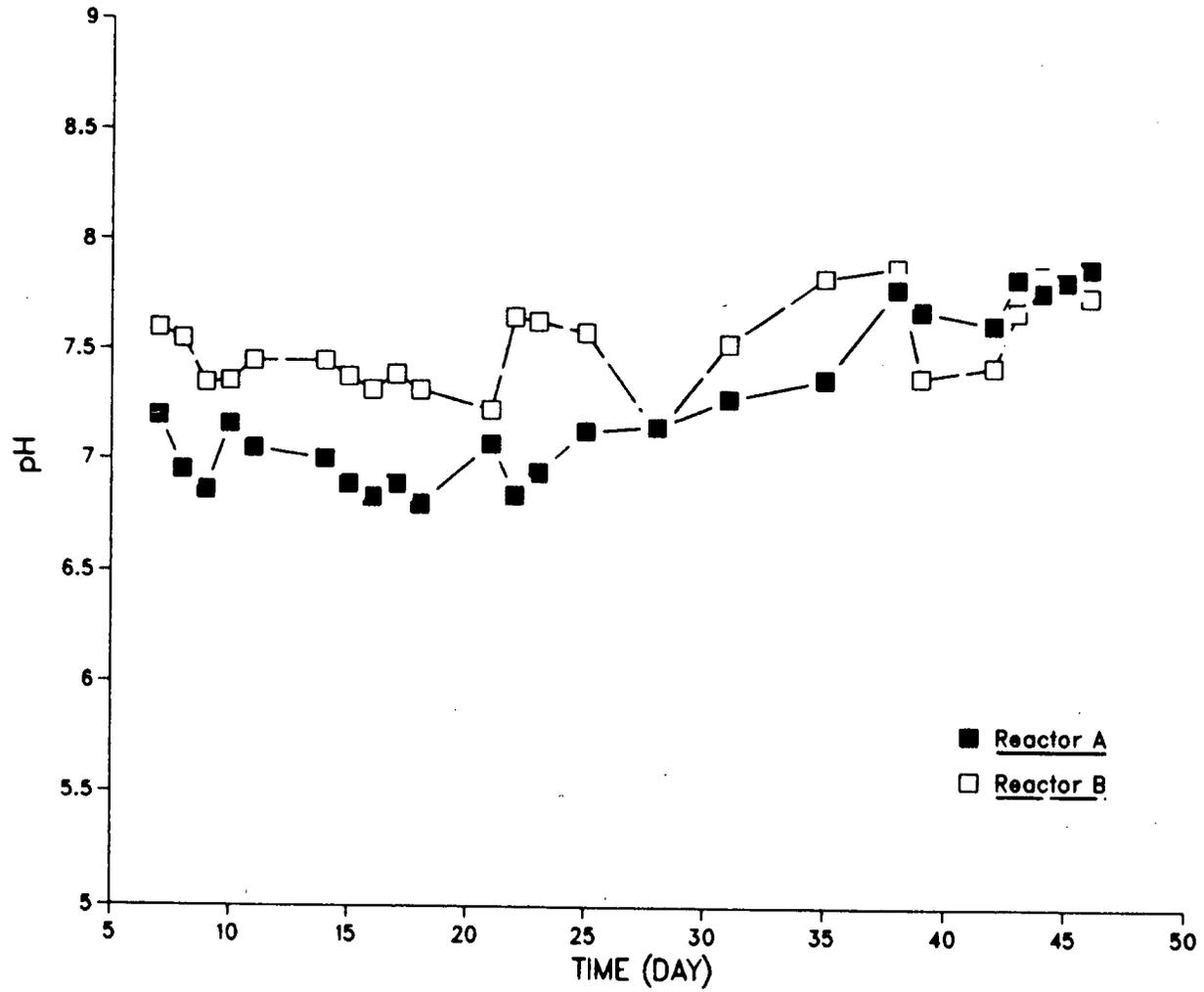


FIGURE 4.12: Effluent pH

# PROPIONIC-ACETIC RATIO

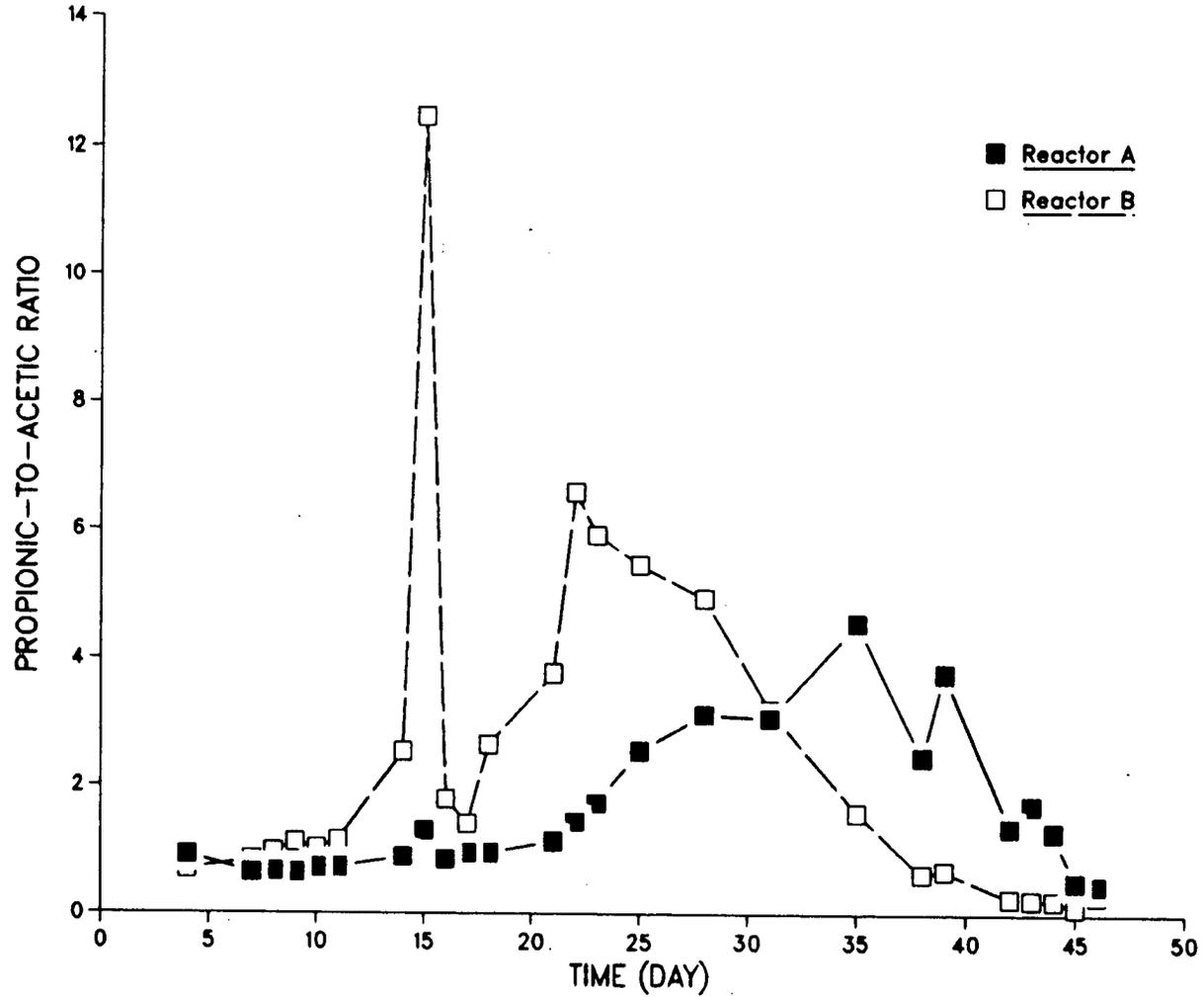


FIGURE 4.13: Effluent Propionic-to-acetic Ratio

Reactor B's ability to reach stability more quickly than reactor A can be attributed to positive effects owing to the installation of ropes in reactor B.

## B. Chapter Conclusion

The important findings and conclusions of the start-up period are presented below:

- The steady-state for the start-up period was reached around day 43 and extended to day 46, the end of the start-up.
- As indicated by Figures 4.2 to 4.9, the first ten to fifteen days of the start-up were characterized by excessive sludge washout because there was enough washed-out sludge in the effluent to offset the COD removed by the bacteria. As microorganisms multiplied and as sludge settleability improved, a negative-to-positive turnover in COD removal was experienced by each reactor.
- It was found that, under similar start-up loading conditions, similar methane yields (utilization efficiencies) could be obtained from vastly different wastewaters like baker's yeast wastewater and cheese whey. In particular, methane yield values of 0.34 and 0.33 (at organic loading rates of 1.11 and 1.27 g COD liter<sup>-1</sup> day<sup>-1</sup>) were achieved in reactors A and B respectively; at similar organic loading rates (0.91 and 1.97 g COD liter<sup>-1</sup> day<sup>-1</sup>), Yan *et al.* (1989a) obtained comparable methane yields of 0.30 and 0.33 with a UASB reactor treating cheese whey at 35°C.
- With regard to the goals of the start-up period, the performance of each reactor can be determined by evaluating the following:
  - 1) How quickly the reactor recovered from excessive sludge washout (timing of turnover);
  - 2) How soon the reactor reached steady-state.
- The advantage of reactor B's fixed-medium in the form of ropes was evident in that, with regard to the Figures 4.1 through 4.9, the negative-to-positive turnover in reactor B always took place sooner than in reactor A.
- The figures in the "START-UP" show that, while the two reactors often displayed a similar behavior pattern, reactor B invariably reached steady-state

more quickly than reactor A. Furthermore, in the course of the start-up period, reactor B was nearly always better than reactor A with respect to methane composition, effluent COD, COD removal, methane yield, effluent VSS and VS, pH, and effluent VFA.

- In some instances, reactor B's steady-state values were not as dramatically different from those of reactor A as had been hoped. An important reason could well be that there had not been enough time for a significantly large amount of biomass to attach to the ropes in reactor B. According to Lo and Liao (1984), it takes 3 to 6 months for biomass to develop on a fixed medium such as acrylic panels. Therefore over a longer period of time, further down the experiment, more substantial advantage in performance should be expected in reactor B.
- Even though raw baker's yeast wastewater contains a high concentration of sulfate (1,700 to 3,200 mg liter<sup>-1</sup>), and even though a run of the same reactors utilizing feed with sulfate removed was not conducted, loss of treatment efficiency in both reactors was believed to be relatively low due to the following reasons: (1) the pH values of the two reactors were rather near the optimum pH range of 7.5 to 8.0 (Sarner et al., 1988); (2) the COD/SO<sub>4</sub><sup>=</sup> ratio of the feed (over 50 g g<sup>-1</sup>) has always been above 10 g g<sup>-1</sup>, which means that natural gas stripping of H<sub>2</sub>S was sufficient to keep H<sub>2</sub>S toxicity down (Lettinga *et al.*, 1985); (3) the seed sludge had already been acclimated to the wastewater prior to being used to seed the reactors; (4) the feed concentration during the start-up period was low (8.0 g COD liter<sup>-1</sup>), which also helped alleviate the sulfate problem (Lettinga *et al.*, 1985).

## V. STEP-UP OPERATION

During the start-up period, the sludge in the reactors was allowed to grow and adapt to the system environment. After the start-up, the reactors were ready to take in feed at higher strengths. This new phase of experimentation began in early March of 1988 and terminated at the end of October of the same year.

Feed strength was increased stepwise (and hence the name "STEP-UP OPERATION") from about 8 to 58 g COD liter<sup>-1</sup>; the major step-up feed concentrations were 18, 25, 30, 35, 40, 45, 50, 55, 58 g COD liter<sup>-1</sup>. As indicated earlier, the start-up feed strength was set at 8 g COD liter<sup>-1</sup>. After the start-up, instead of raising feed strength abruptly to, say, 40 g COD liter<sup>-1</sup>, a gradual increase was adopted for the following reasons:

- The reactors immediately after the start-up period were specially sensitive to environmental factors such as the organic loading rate. An excessively high organic loading rate would cause too much buoyancy due to the produced biogas and result in a high rate of sludge washout. At this stage of the experiment, special care must be taken to minimize sludge washout; otherwise, stable conditions might be unduly delayed or never reached (Barford, 1988; Wu *et al.*, 1985).
- The stepwise feed strength increase not only allowed the microorganisms to accustom themselves to the new environment, but also permitted a full view of the performance profile of the reactors over the entire range of the operational settings. Moreover, the researcher had the advantage of locating the optimum setting as well as observing the transient response of the system to the change of the operational parameters. In the case of locating the optimum-performance point, if the feed strength increment was too great, the optimum point might very well fall in the middle of the increment interval; then either the researcher would not be able to detect the presence of such an optimum point, or it would be very hard to determine with reasonable accuracy the specifics of the optimum setting. If in fact an optimum point had been missed, attempt to run the reactors "backwards" (i.e., to run them at a lower feed strength) would most likely not lead to the optimum point of interest because of the phenomenon called hysteresis. Hysteresis is explained in greater detail in a later section, but for now it suffices to say that, in the context of this study, the

pathway along which the reactor was operated during the step-up operation would often be different from the pathway followed when the reactor was run with decreasing (step-down) feed strength.

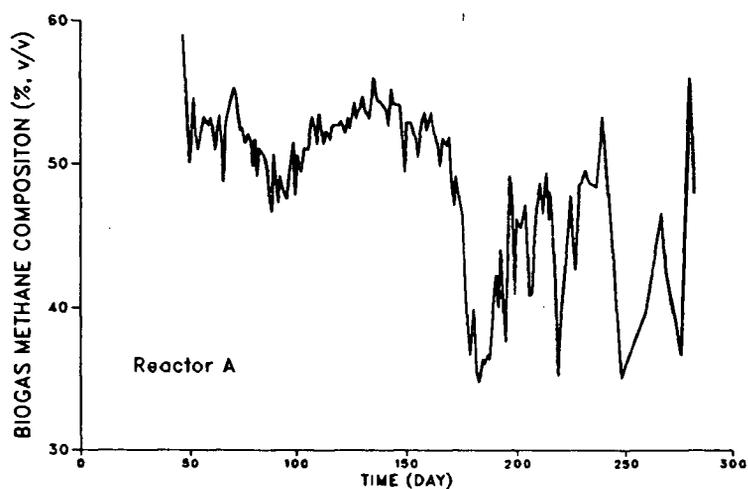
## A. Results and Discussions

### 1) Steady-state and Transient Analysis

The steady-state definition for the step-up operation period differs from that of the start-up period. Whereas steady state in the start-up period represented the time span in which system parameter variations were within 10%, the steady-state value of a system parameter (e.g., methane composition) in the step-up period was defined as the arithmetic mean of that parameter over the time period in which the operating conditions were kept constant and the reactors were fed with the same batch of feed (that is, same organic loading rate). An average parameter value calculated in this fashion was considered to yield a reasonable steady-state value since a perfectly "horizontal" steady-state would be most unlikely in the UASB reactor, where the amount of sludge and its activity are constantly changing and more dynamic than in batch reactors.

For the transient analysis, because of the large number of data points collected in the experiment, it was felt best to work with the constant-organic-loading averages obtained by the method outlined above. (For completeness' sake, however, two sample time plots depicting *every* data point can be found in Figure 5.0.) Therefore, the transient analysis presented in this study was actually based on points which were themselves averages. The effect of this practice was that day-to-day variation within any given constant-loading period was no longer visible. In effect the transient analysis was depicting the transient behavior of individual constant-loading averages over the experiment time domain. One would think that there was not much of a difference between the steady-state and transient analyses. However, a fundamental

### BIOGAS METHANE COMPOSITION TIME PLOT



### BIOGAS METHANE COMPOSITION TIME PLOT

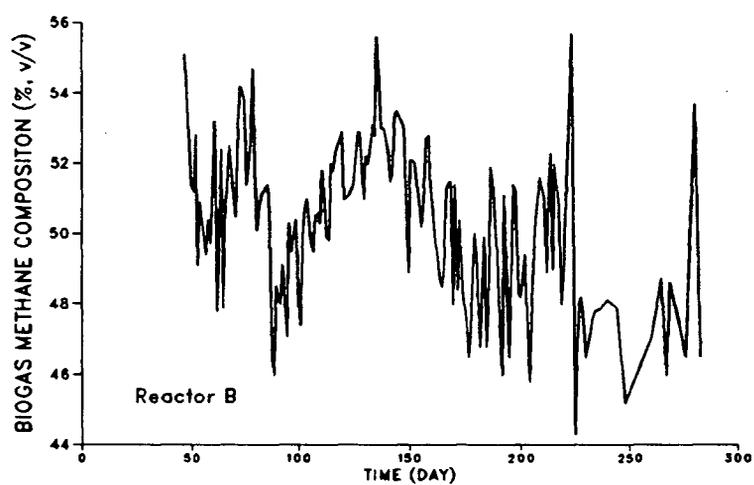


Figure 5.0: Time plot of step-up operation methane composition of reactors A and B

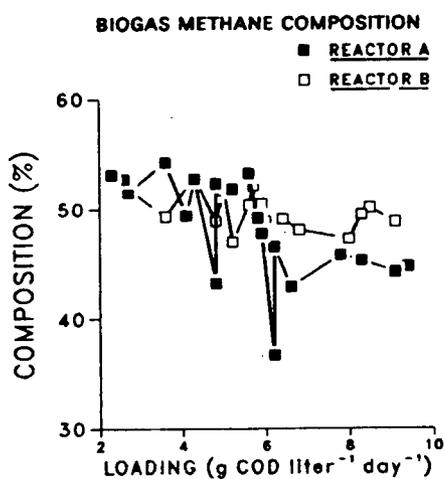
difference did exist between the two analyses. The steady-state analysis focused on the average values of the parameters of interest in relation to *organic loading rates*, since all other parameters were kept constant. The transient analysis, in contrast, was interested in the trend of the same averages in relation to the *experiment time*.

The steady-state and transient results in terms of biogas methane composition (% by volume), methane production (liter CH<sub>4</sub> liter<sup>-1</sup> day<sup>-1</sup>), effluent COD (g COD liter<sup>-1</sup>), COD reduction (%), and methane yield (liter CH<sub>4</sub> g<sup>-1</sup> COD day<sup>-1</sup>) are summarized in Table 5.1 and depicted in Figures 5.1 and 5.2. Table 5.1 presents the data chronologically, from day 47 (which is the first day following the end of the start-up period) to day 268 which marks the beginning of the last constant-loading period of the entire experiment. Figure 5.1 is based on organic loading rates (g COD liter<sup>-1</sup> day<sup>-1</sup>) sorted in the ascending order, and Figure 5.2 on days of the experiment as the x axis. All the values used are arithmetic averages for individual constant-loading periods.

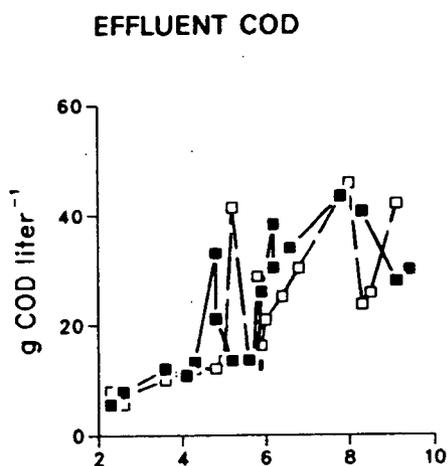
### ***a) Biogas Methane Composition***

As can be seen in Figure 5.1a, the two reactors displayed a descending pattern in biogas methane composition. This pattern is in keeping with the fact that, in general, the biogas methane composition tends to fall with increasing organic loading.

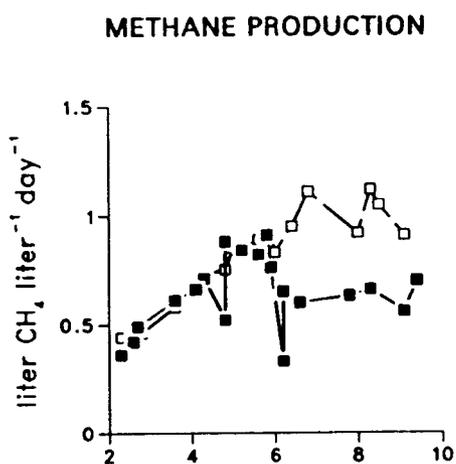
Both reactors started out with a methane composition of around 53% (volume basis) at an organic loading rate of 2.3 g COD liter<sup>-1</sup> day<sup>-1</sup>. However, at organic loading rates higher than 6 g COD liter<sup>-1</sup> day<sup>-1</sup>, reactor A's methane composition oscillated around 45% while reactor B's was around 49%. In other words, whereas reactor A experienced a significant drop in methane composition (53% down to 45%), reactor B's methane composition went through little decrease. Furthermore, reactor A's methane composition fluctuated much more than that of reactor B. Reactor B's



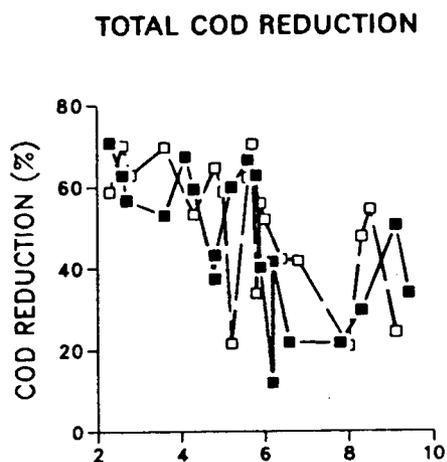
(a)



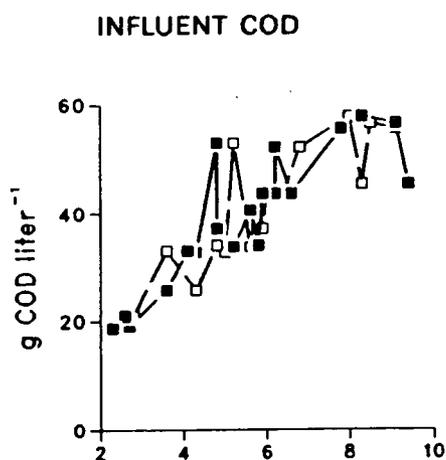
(d)



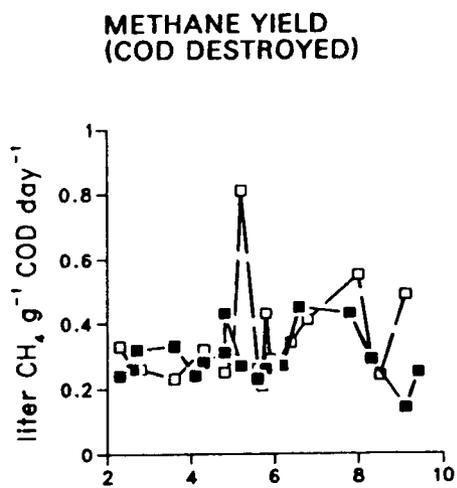
(b)



(e)

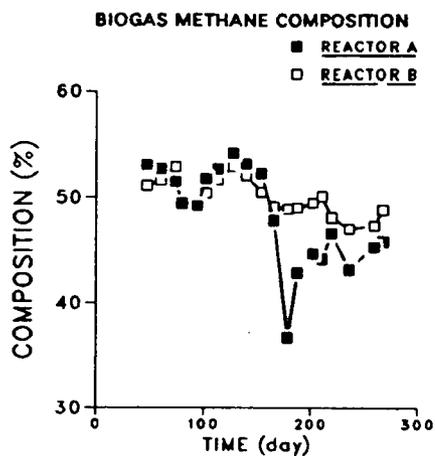


(c)

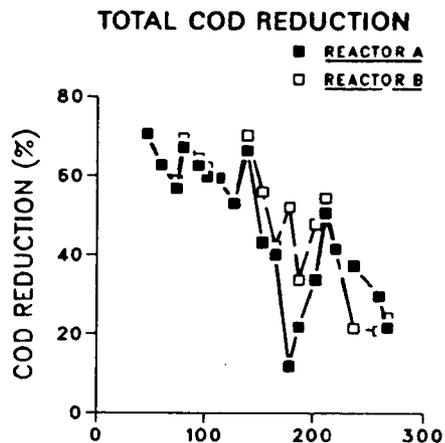


(f)

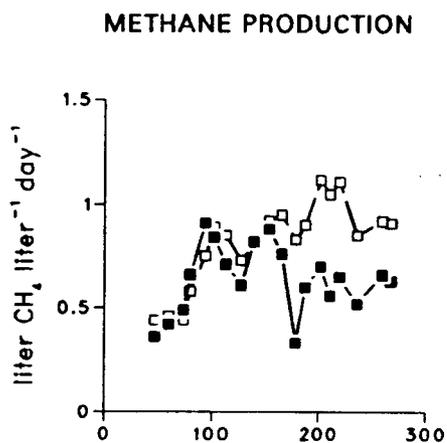
FIGURE 5.1: Treatment Efficiency versus Organic Loading



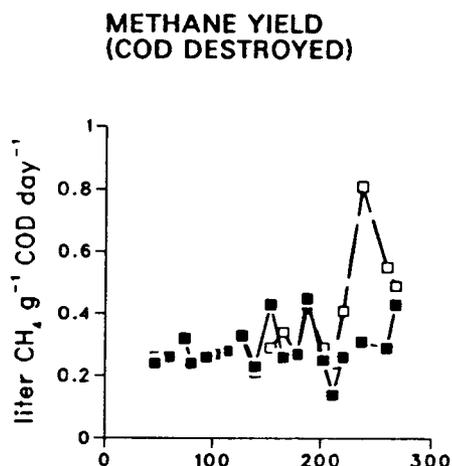
(a)



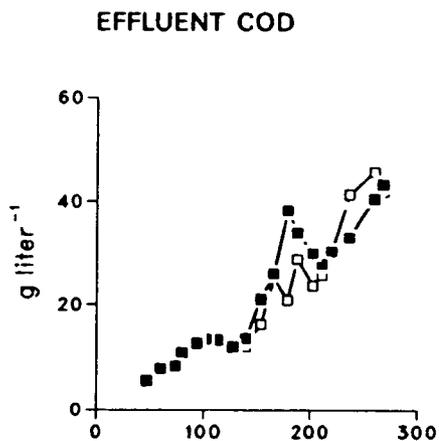
(d)



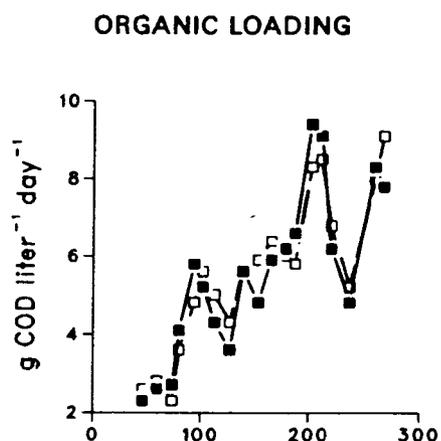
(b)



(e)



(c)



(f)

FIGURE 5.2: Treatment Efficiency versus Time

stability was believed to be due to the installed fixed-film structure (Lo *et al.*, 1984; Vega *et al.*, 1988).

**TABLE 5.1: Step-up operation treatment efficiency\***

Day	Feed	Effluent		COD		average		Organic		Methane		Yield		Yield	
		COD	COD	Reduction	Reduction	CH <sub>4</sub> %	CH <sub>4</sub> %	Loading	Loading	Production	Production	COD Added	COD Added	COD Dest.	COD Dest.
		A	B	A	B	A	B	A	B	A	B	A	B	A	B
47	18.6	5.5	5.6	70.7	70.0	53.1	51.1	2.3	2.6	0.36	0.44	0.16	0.17	0.24	0.26
60	20.9	7.8	7.7	62.8	62.9	52.7	51.6	2.6	2.8	0.42	0.46	0.16	0.17	0.26	0.26
74	19.1	8.3	7.9	56.8	58.8	51.5	52.9	2.7	2.3	0.49	0.44	0.18	0.19	0.32	0.33
80	33.1	10.8	10.0	67.3	69.6	49.4	49.3	4.1	3.6	0.66	0.58	0.16	0.16	0.24	0.23
94	34.1	12.7	12.1	62.7	64.6	49.2	48.9	5.8	4.8	0.91	0.75	0.16	0.16	0.26	0.25
102	33.8	13.5	12.8	59.9	62.3	51.8	50.4	5.2	5.6	0.84	0.89	0.16	0.16	0.27	0.26
113	32.8	13.3	13.5	59.5	58.9	52.7	51.7	4.3	5.0	0.71	0.85	0.16	0.17	0.28	0.29
127	25.6	12.0	12.0	53.1	53.3	54.2	52.9	3.6	4.3	0.61	0.73	0.17	0.17	0.33	0.32
139	40.6	13.6	12.1	66.5	70.3	53.2	52.1	5.6	5.7	0.82	0.81	0.15	0.15	0.23	0.21
153	37.2	21.1	16.3	43.2	56.1	52.3	50.5	4.8	5.9	0.88	0.92	0.19	0.16	0.43	0.29
165	43.6	26.0	25.1	40.2	42.3	47.8	49.1	5.9	6.4	0.76	0.95	0.13	0.15	0.26	0.34
178	43.6	38.3	20.9	12.0	52.1	36.7	48.9	6.2	6.0	0.33	0.83	0.06	0.14	0.27	0.27
187	43.6	34.0	28.8	21.9	33.8	42.9	49.0	6.6	5.8	0.60	0.90	0.09	0.16	0.45	0.43
202	45.3	30.0	23.6	33.9	47.8	44.7	49.5	9.4	8.3	0.70	1.12	0.08	0.14	0.25	0.29
211	56.5	27.9	25.7	50.6	54.5	44.2	50.1	9.1	8.5	0.56	1.05	0.07	0.13	0.14	0.24
220	52.1	30.4	30.3	41.6	41.8	46.6	48.1	6.2	6.8	0.65	1.11	0.12	0.16	0.26	0.41
237	52.9	33.1	41.4	37.4	21.6	43.2	47.0	4.8	5.2	0.52	0.85	0.11	0.17	0.31	0.81
260	57.8	40.6	45.7	29.7	20.9	45.3	47.3	8.3	8.0	0.66	0.92	0.08	0.11	0.29	0.55
268	55.5	43.4	42.0	21.7	24.3	45.8	48.8	7.8	9.1	0.63	0.91	0.10	0.11	0.43	0.49

\* Feed and effluent COD expressed in g liter<sup>-1</sup>; COD reduction and methane composition in %; organic loading in g COD liter<sup>-1</sup> day<sup>-1</sup>; methane production in liter CH<sub>4</sub> liter<sup>-1</sup> day<sup>-1</sup>; yields in liter CH<sub>4</sub> g<sup>-1</sup> COD day<sup>-1</sup>, either added or destroyed.

The maximum biogas methane composition obtained in the step-up operation phase was 54% (at an organic loading rate of 3.6 g COD liter<sup>-1</sup> day<sup>-1</sup>) and was comparable with that reported by Frostell (1982) who achieved a maximum biogas methane composition of 56% (at an organic loading rate of 22 g COD liter<sup>-1</sup> day<sup>-1</sup>) from a laboratory-scale continuously fed anaerobic fluidized bed reactor treating baker's yeast wastewater at 30°C. Lo *et al.* (1989), using an anaerobic rotating biological contactor (ARBC) reactor with an active biomass, achieved a maximum methane composition of 48% at a loading rate of 9.2 g COD liter<sup>-1</sup> day<sup>-1</sup>. As indicated earlier,

the maximum methane composition achieved in the start-up period was 59% (at an organic loading rate of 1.1 g COD liter<sup>-1</sup> day<sup>-1</sup>). These figures seem to suggest that the maximum biogas methane composition achievable with baker's yeast wastewater is 60%.

Based on the actual experiment time, Figure 5.2a shows the transient behavior of the reactors. It can be seen that both reactors oscillated around 50% of methane content for the first 165 days. Thereafter, each reactor took on a different pattern: reactor B's methane composition experienced a slight global drop while going through some local mild oscillation; reactor A, however, began to go down a deep valley and would not start to recover partially until after day 178. It is clear that, as the organic loading began to rise, the increasing stress on the reactors also began to be reflected in the lowering of methane composition, especially after day 168. In particular, when the organic loading rate rose to about 6 g COD liter<sup>-1</sup> day<sup>-1</sup> (day 168), the fixed-medium in reactor B started to prove advantageous.

### ***b) Volumetric Methane Production***

For the same organic loading range of 2.3 to 9.4 g COD liter<sup>-1</sup> day<sup>-1</sup>, reactor A's methane production rate (based on standard temperature and pressure, STP, 0°C and one atmosphere) ranged between 0.34 and 0.91 liter CH<sub>4</sub> liter<sup>-1</sup> day<sup>-1</sup> while that of reactor B ranged between 0.44 and 1.12 liter CH<sub>4</sub> liter<sup>-1</sup> day<sup>-1</sup> (Figure 5.1b).

Based on methane production over the entire experiment, reactor B definitely was better than reactor A. Reactor A's methane production rate rose from 0.36 liter CH<sub>4</sub> liter<sup>-1</sup> day<sup>-1</sup> at the loading of 2.3 g COD liter<sup>-1</sup> day<sup>-1</sup> and attained its maximum of 0.91 liter CH<sub>4</sub> liter<sup>-1</sup> day<sup>-1</sup> at the loading of 5.8 g COD liter<sup>-1</sup> day<sup>-1</sup>; then it dropped abruptly and stayed at around 0.7 liter CH<sub>4</sub> liter<sup>-1</sup> day<sup>-1</sup>. Unlike reactor A, reactor B's methane production curve was essentially ascending throughout the whole organic

loading range. The maximum methane production rate achieved by reactor B was 1.12 liter  $\text{CH}_4$  liter<sup>-1</sup> day<sup>-1</sup> at the loading of 8.3 g COD liter<sup>-1</sup> day<sup>-1</sup>. Obviously, reactor A had already achieved its maximum volumetric gas production at an organic loading rate (5.8 g COD liter<sup>-1</sup> day<sup>-1</sup>) which was still far from the highest loading rate (9.4 g COD liter<sup>-1</sup> day<sup>-1</sup>) imposed in this study. Reactor B, however, did not appear to have reached its highest-possible gas production even at the maximum loading applied to it (9.1 g COD liter<sup>-1</sup> day<sup>-1</sup>) since it is not clear whether the curve would drop or continue to rise. Higher organic loading rates would have to be applied to determine the organic loading rate above which reactor B's gas production would begin to decrease. Furthermore, whereas reactor A's methane production rate fluctuated sharply between the loading range of 4 and 7 g COD liter<sup>-1</sup> day<sup>-1</sup>, reactor B's was rather smooth. Only for loading rates higher than 7 g COD liter<sup>-1</sup> day<sup>-1</sup> did reactor B experience some bumpiness in methane production.

The methane production rate trend in Figure 5.2b, aside from the difference in value between the two reactors, is consistent with the fact that methane production rate follows the organic loading rate closely. That is, the methane production rate rises as the loading rises, and vice versa. The exception is the deep valley in reactor A's gas production curve, which represents that period of time (the bottom of the valley is located at day 178 in Figure 5.2b) when reactor A is still in the process of adapting to the new higher organic loading rate (6 g COD liter<sup>-1</sup> day<sup>-1</sup>). Evidently, the additional biomass clung to the ropes enhanced reactor B's biogas production, both quantitatively and qualitatively, and at the same time rendered reactor B more tolerant to organic loading rates higher than 6 g COD liter<sup>-1</sup> day<sup>-1</sup>.

By comparison, an ARBC reactor treating baker's yeast wastewater at 35°C produced only 0.61 liter  $\text{CH}_4$  liter<sup>-1</sup> day<sup>-1</sup> at a comparable loading of 6.5 g COD liter<sup>-1</sup> day<sup>-1</sup> (Lo *et al.*, 1988). At a loading of 9.2 g COD liter<sup>-1</sup> day<sup>-1</sup>, the same ARBC

reactor produced 1.17 liter  $\text{CH}_4$  liter<sup>-1</sup> day<sup>-1</sup> (Lo et al., 1989), as compared to 0.70 and 0.91 liter  $\text{CH}_4$  liter<sup>-1</sup> day<sup>-1</sup> for reactors A and B respectively at a similar loading. The better gas production by reactors A and B at the intermediate loading rates is largely due to the fact that the UASB reactor is able to retain sludge that would otherwise be lost in the effluent, and to minimize the negative effects of shock loading by virtue of continuous feeding. (Bacteria have more time to assimilate the incoming feed when it is continuous rather than batch feeding. It appears that digestion efficiency is higher when continuous feeding is employed— Kennedy and van den Berg, 1982.) At loading rates over 9 g COD liter<sup>-1</sup> day<sup>-1</sup>, both reactors' gas production rates were less than that achieved by the ARBC reactor. A possible explanation is that the ARBC reactor had the advantage of possessing a bacteria population that had already been well established prior to the experiment, whereas reactors A and B were being run for the first time and were under much loading stress.

Sayed *et al.* (1987) treated slaughterhouse wastewater using a granular sludge UASB reactor run at 30°C. The wastewater contained 40-50% of coarse suspended matter and a COD-to-BOD ratio of 3 to 4, which was comparable with that of baker's yeast wastewater. At a loading of 6.4 g COD liter<sup>-1</sup> day<sup>-1</sup>, a methane production rate of 1.11 liter  $\text{CH}_4$  liter<sup>-1</sup> day<sup>-1</sup> was achieved, as compared with 0.9 liter  $\text{CH}_4$  liter<sup>-1</sup> day<sup>-1</sup> attained by reactors A and B at 5.8 g COD liter<sup>-1</sup> day<sup>-1</sup>. While Sayed's result was relatively close to that achieved by reactors A and B at the loading of about 6 g COD liter<sup>-1</sup> day<sup>-1</sup>, at an organic loading rate of approximately 10.0 g COD liter<sup>-1</sup> day<sup>-1</sup>, however, the methane production rate achieved by Sayed's reactor was 1.5 liter  $\text{CH}_4$  liter<sup>-1</sup> day<sup>-1</sup>, much higher than those of reactors A and B ( 0.70 and 0.91 liter  $\text{CH}_4$  liter<sup>-1</sup> day<sup>-1</sup> respectively). Some possible explanations follow. First, granular sludge, being more active than the mostly muddy sludge observed in reactors A and B, led to a higher gas output. Second, while similar organic loading rates were applied,

the wastewater used in this study was much more concentrated in COD than that of the wastewater treated by Sayed— 8 to 58 g COD liter<sup>-1</sup> as compared to 1.5 to 2.2 g COD liter<sup>-1</sup>. The UASB reactor is normally used to treat wastewaters of rather low COD concentrations (typically between 1 and 20 g COD liter<sup>-1</sup>), such as municipal sewage wastewater and sugar-beet sap at relatively high hydraulic loading rates (Lettinga *et al.*, 1980 and 1985). Moreover, as mentioned in the literature review chapter, the UASB reactor requires a certain selection pressure (in the form of controlled high hydraulic loading) so as to drive out less healthy sludge and reserve more available nutrient for the larger sludge aggregates to grow more rapidly (Wu *et al.*, 1985; Lettinga *et al.*, 1985). Conceivably, Sayed's better results might be due to a better selection pressure obtained through more vigorous hydraulic loading rates. In this study, however, it has been stated earlier that ensuring stable operation and achieving truly continuous feeding (twenty-four hours a day, seven days a week) to minimize loading shock were the reasons for the relatively low hydraulic loading rate applied throughout the entire experiment (2.0 to 2.5 liter of feed per day).

### ***c) COD Reduction***

Reactors A and B displayed the same diminishing general trend in COD reduction as indicated by Figures 5.1e and 5.2d. The overall pattern for the two reactors is again within expectation: COD removal deteriorates with rising organic loading.

It is worth noting that, while reactor B achieved more removal than reactor A from days 139 to 220, the scenario reversed towards the higher end of organic loading (i.e., 8 g COD liter<sup>-1</sup> day<sup>-1</sup> and up). In this high loading range, relatively large amounts of washed-out sludge were observed for reactor B— though to a lesser extent than for reactor A. This excessive sludge washout is thought to be the same sludge

washout phenomenon that reactor A had experienced earlier (beginning from day 150); only it is a "lagged" one in the case of reactor B.

A careful examination of COD reduction and volumetric gas production shed more light on the time lag observed in reactor B. Figure 5.2b shows that following day 150, reactor A's gas production dropped considerably, but reactor B's gas production climbed drastically in relation to reactor A and amounted to over 1.0 liter  $\text{CH}_4$  liter<sup>-1</sup> day<sup>-1</sup> by day 220. In this experiment, Flotation, and not turbulence, was thought to be the main mechanism responsible for the buoyancy of sludge flocs. Higher gas production means more flotation of the sludge due to gas bubbles, which results in a higher sludge loss rate (Lettinga *et al.*, 1980 and 1985). Thus, reactor A's drop in gas production effected a lowering in sludge washout (relative to before day 150); in contrast, reactor B experienced a surge of gas production which obviously caused the excessive sludge loss in reactor B.

Since baker's yeast wastewater is rather slow to biodegrade, as indicated by its high COD-BOD ratio, the maximum COD reduction achieved in this study (71% at 2.3 g COD liter<sup>-1</sup> day<sup>-1</sup>) was still low compared to that of more readily biodegradable wastewaters such as cheese whey, which is very readily biodegradable (Yan *et al.*, 1989a). However, at organic loading rates higher than 6 g COD liter<sup>-1</sup> day<sup>-1</sup> (after day 150), COD reduction fell below 50% for both reactors; single-stage treatment at this efficiency in this loading range was considered rather unattractive. Higher dilution of the original wastewater, recycle of the effluent, and/or longer retention time would be required to achieve desired COD removal.

#### ***d) Methane Yield***

From Figure 5.1f, up to organic loading rates of 6 g COD liter<sup>-1</sup> day<sup>-1</sup>, the methane yield (based on COD destroyed) for both reactors was mostly bracketed in the

range of 0.25 to 0.35 liter  $\text{CH}_4 \text{ g}^{-1} \text{ COD day}^{-1}$ . At loading rates higher than 6 g COD liter $^{-1} \text{ day}^{-1}$  reactor A's methane yield oscillated between 0.15 and 0.45 liter  $\text{CH}_4 \text{ g}^{-1} \text{ COD day}^{-1}$  whereas reactor B's between 0.25 and 0.50 liter  $\text{CH}_4 \text{ g}^{-1} \text{ COD day}^{-1}$ . Clearly, as organic loading increased, contribution to methane yield from reactor B's fixed-medium also began to stand out.

Time-wise, the methane yield looked essentially the same for the two reactors for up to day 200 (Figure 5.2e). After this time, the two reactors began to differentiate between themselves. Reactor A went through a slight valley and then levelled off towards the end of the experiment. As to reactor B, instead of going through a valley as reactor A, it reached a methane yield of 0.81 liter  $\text{CH}_4 \text{ g}^{-1} \text{ COD day}^{-1}$ ; then it subsided and nearly merged with reactor A at the very end.

By comparison, also treating baker's yeast wastewater from the same source, Lo *et al.* (1989) reported a methane yield of 0.28 liter  $\text{CH}_4 \text{ g}^{-1} \text{ COD day}^{-1}$  at a loading of 9.1 g COD liter $^{-1} \text{ day}^{-1}$ . At similar loading rates of 9.4 and 8.3 g COD liter $^{-1} \text{ day}^{-1}$  (taken from the period starting at day 202), the two reactors achieved methane yields of 0.25 and 0.29 liter  $\text{CH}_4 \text{ g}^{-1} \text{ COD day}^{-1}$  respectively. Clearly, whereas reactor A proved to be no better than Lo's batch reactor, the biomass support system indeed had a positive impact on reactor B's methane yield. Furthermore, Yan *et al.* (1989a), treating cheese whey in a UASB using a similar setup and operating conditions, observed rather consistent methane yield of 0.30 to 0.34 liter  $\text{CH}_4 \text{ g}^{-1} \text{ COD day}^{-1}$  for a loading range of 0.91 to 7.77 g COD liter $^{-1} \text{ day}^{-1}$ . By and large, the results obtained in this study were comparable with those of Yan *et al.* (1989a)— even though cheese whey and baker's yeast wastewater are rather different in nature.

### *e) Volatile Fatty Acids*

The well-being of the anaerobic reactor can largely be explained by its VFA levels. Therefore by closely following the VFA concentrations of the reactor, one can determine whether digestion is proceeding smoothly or process failure is imminent. As indicated in the "LITERATURE REVIEW", TVFA greater than 2000 mg liter<sup>-1</sup>, acetic acid higher than 800 mg liter<sup>-1</sup>, and a P/A ratio greater than 1.4 were reliable methods of predicting the failure of anaerobic process. Of these criteria, the combination of acetic acid and the propionic-to-acetic-acid ratio provides the best index for digestion failure prediction. Additionally, long-chain VFA's, butyric and iso-butyric acids, also give useful indication as to the extent of stress an anaerobic reactor is under. Iso-butyric acid levels of 5, 5-15, above 15 mg liter<sup>-1</sup> indicate that the reactor is, respectively, under little or no stress, under much stress and near failure, is failing.

VFA data are summarized in Table 5.2. Up until day 165, TVFA for both reactors remained below 500 mg liter<sup>-1</sup> (Figure 5.3a). From day 165 onward, TVFA levels rose rapidly, especially for reactor A. By day 187 reactor A's TVFA had already gone beyond the 2,000 mg liter<sup>-1</sup> level, never to subside again; eventually it exceeded 4,000 mg liter<sup>-1</sup>. Reactor B, however, always stayed below the 2,000 mg liter<sup>-1</sup> mark throughout the experiment, except towards the very end when the TVFA amounted to 2,500 mg liter<sup>-1</sup>. With particular reference to the TVFA failure criterion alone, the following points can be drawn:

- The first 165 days were a period in which both reactors were under little, if any, stress.
- During the subsequent period, from days 165 to 187, increasing stress on both reactors, especially A, manifested in the form of steep TVFA increase.
- From day 187 to the end of the experiment was the final period, in which failure began to set in for reactor A which was now characterized by TVFA concentrations well beyond 2000 mg liter<sup>-1</sup> and low treatment efficiency.

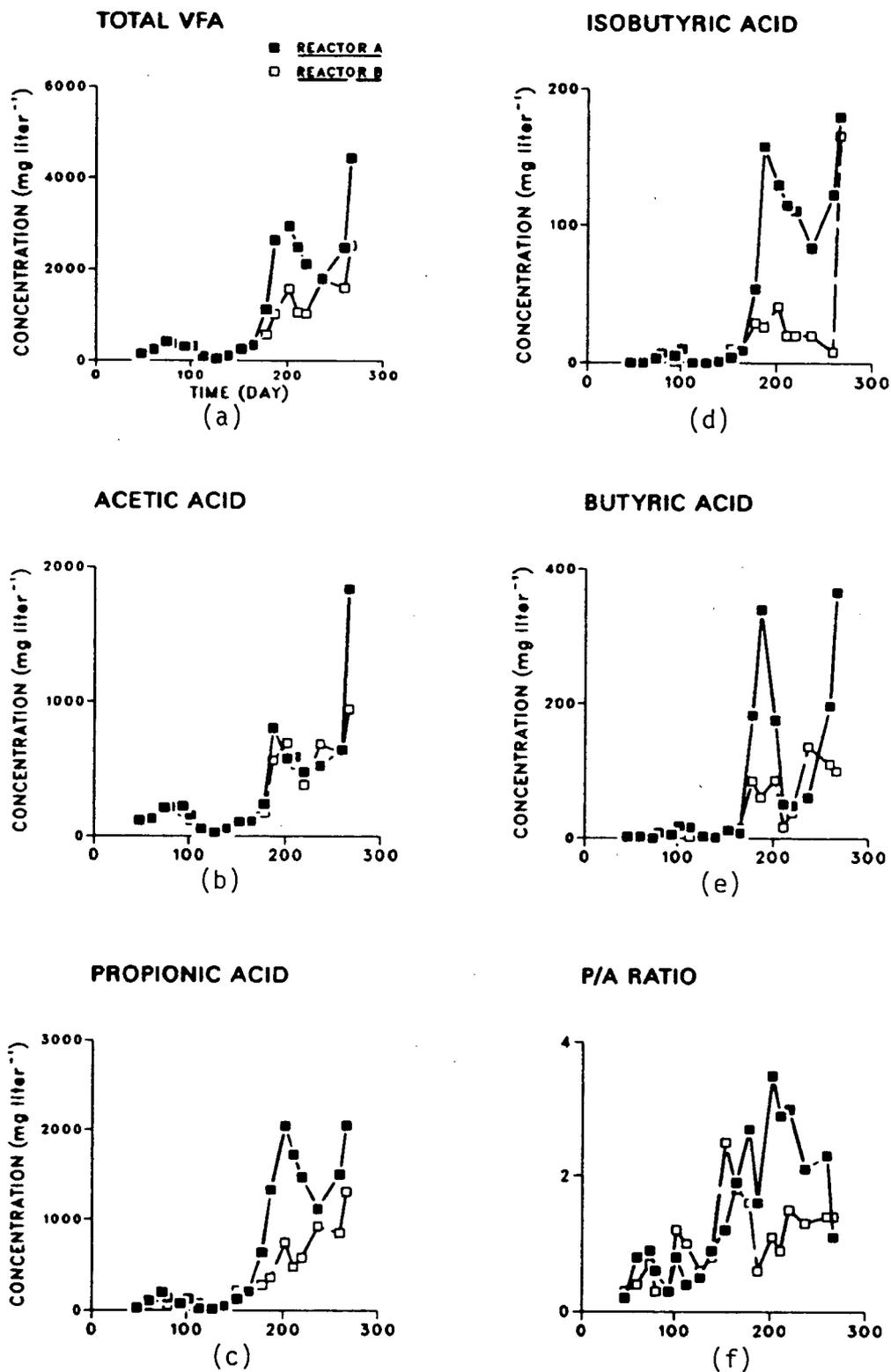


FIGURE 5.3: Volatile Fatty Acids versus Time

Reactor B, however, was under a lot of stress but never did approach the point of failure until after day 267.

Figure 5.3b shows that, except after day 267, the acetic acid levels of the two reactors were virtually always under 800 mg liter<sup>-1</sup>. If failure prediction was solely based on the failure criterion of acetic acid, then the implication was that failure would not come to any of the reactors until after day 267. However, this statement is not in harmony with the preceding discussion on TVFA. The other failure criteria have to be checked before it is possible to make any valid conclusions.

**TABLE 5.2: Volatile fatty acids of the step-up operation**

Day	Acetic <sup>1</sup>		Propionic		Isobutyric		Butyric		TVFA		P/A		Loading <sup>2</sup>	
	A	B	A	B	A	B	A	B	A	B	A	B	A	B
47	118	96	26	34	0	0	2	0	146	130	0.2	0.3	2.3	2.6
60	130	127	105	54	0	0	2	0	237	181	0.8	0.4	2.6	2.8
74	212	207	200	138	3	0	0	0	415	345	0.9	0.7	2.7	2.3
80	215	225	138	59	7	3	8	4	368	291	0.6	0.3	4.1	3.6
94	224	180	74	49	5	1	5	2	309	232	0.3	0.3	5.8	4.8
102	156	115	127	135	10	4	18	14	310	268	0.8	1.2	5.2	5.6
113	56	72	22	75	0	2	16	2	94	150	0.4	1.0	4.3	5.0
127	28	49	13	31	0	0	3	1	44	81	0.5	0.6	3.6	4.3
139	57	66	49	54	1	3	1	3	107	126	0.9	0.8	5.6	5.7
153	106	87	127	219	4	10	12	9	248	324	1.2	2.5	4.8	5.9
165	111	107	209	189	9	11	8	15	338	322	1.9	1.8	5.9	6.4
178	240	172	640	278	54	29	183	86	1117	566	2.7	1.6	6.2	6.0
187	806	566	1329	363	158	26	340	61	2633	1016	1.6	0.6	6.6	5.8
202	582	694	2045	746	130	41	176	87	2933	1568	3.5	1.1	9.4	8.3
211	593	534	1719	480	115	20	51	17	2478	1051	2.9	0.9	9.1	8.5
220	482	384	1469	581	111	20	49	38	2112	1022	3.0	1.5	6.2	6.8
237	526	689	1119	927	84	20	61	137	1791	1774	2.1	1.3	4.8	5.2
260	647	622	1498	850	123	8	197	111	2465	1592	2.3	1.4	8.3	8.0
267	1839	945	2055	1306	180	166	366	101	4441	2517	1.1	1.4	7.8	9.1

<sup>1</sup> All VFA's expressed in mg liter<sup>-1</sup>.

<sup>2</sup> Units: g COD liter<sup>-1</sup> day<sup>-1</sup>.

The P/A ratio is defined as the ratio of propionic and acetic acids, which are shown in Figures 5.3b, 5.3c, and 5.3f. Before day 153, P/A ratios for both reactors were by and large under 1.4. Thereafter, as stress built up, the P/A ratio of reactor A

always stayed greater than 1.4. Unlike reactor A, after day 153, reactor B's P/A ratio consistently remained below 1.4 until the termination of the study— though it is rather near 1.4. Based on the P/A ratio alone, it can be said that, after day 153, reactor A was under much stress and failure very likely started to set in sometime after day 187— as evidenced by reactor A's ever-increasing P/A ratio (Figure 5.3f). As to reactor B, it never did reach failure because its P/A ratio always under 1.4.

Always found in much smaller concentrations than acetic and propionic acids, iso-butyric and butyric acids provide additional information concerning digestion failure. Since Hill *et al.* (1987 and 1988) showed that the iso-forms of long-chain VFA's are particularly useful, the discussion will center around iso-butyric acid (Figure 5.3d); the butyric acid graph (Figure 5.3e) is included here for completeness' sake. The butyric acid levels of the two reactors were mostly below 10 mg liter<sup>-1</sup> up to day 165; thereafter, however, it sky-rocketed abruptly, especially for reactor A, to well above the 5-15 mg liter<sup>-1</sup> criterion range. Based on the iso-butyric failure criterion, reactor A was well into failure after day 187. Meanwhile, reactor B was just marginally over the 15 mg liter<sup>-1</sup> mark in iso-butyric acid, declining slightly towards the end of the experiment. Except after day 267 when the iso-butyric acid went up to 166 mg liter<sup>-1</sup>, it is felt that reactor B did not really fail in spite of its iso-butyric acid levels being greater than 15 mg liter<sup>-1</sup>. Reactor B was on the "border line".

After examining each failure criterion, some conclusions may now be drawn:

- All failure criteria pointed to the same conclusion: the first 153 days of the experiment were marked by stability (low concentrations for all VFA's) which is also confirmed by Figures 5.1 and 5.2 in the previous sections.
- Except for the acetic failure criterion, all other criteria agreed that, sometime after day 153 and before day 165, stress levels began to increase drastically for both reactors— especially reactor A. In the case of the acetic acid, it is conceivable that this acetic acid criterion value (800 mg liter<sup>-1</sup>) varies from waste to waste, and from system to system. In this study, Figure 5.3b

suggests that the failure acetic acid level could be somewhere below 800 mg liter<sup>-1</sup>, possibly around 500 mg liter<sup>-1</sup>. If this speculation were true, then it would be hard to explain the reason why there was practically no difference between the two reactors from days 187 to 267.

- Again, except for acetic acid, all criteria indicated that failure began to set in for reactor A after day 187; reactor A's VFA levels exceeded all failure criterion values. For reactor B, all VFA concentrations were lower than the failure criterion values. The exception was iso-butyric acid; it slightly exceeded the criterion value between days 165 and 267. In addition, reactor B's iso-butyric acid reached a high of 166 mg liter<sup>-1</sup> at the very end. The marginally high iso-butyric levels and the *single* peak at day 267 (166 mg liter<sup>-1</sup>) were not evidence enough to substantiate the failure of reactor B— since all other failure criteria had not been exceeded. Thus, the best conclusion would be that, after day 165, reactor B experienced much stress and *approached* failure towards the end of the experiment.
- In summary, the VFA results showed that reactor B performed better than reactor A. As of now, all that can be said about the reason of such superiority on reactor B's part is that the combined (or total) bacterial activity of reactor B was higher than that of reactor A; a look at the reactor's behavior along the height of the reactor (i.e., profile study) will shed further light not only on the performance of each individual reactor but the reason for the difference between them.

### *f) Sulfate Toxicity*

The raw baker's yeast wastewater used in the experiment contained a high concentration of sulfate (1,700 to 3,200 mg liter<sup>-1</sup>) which encourages sulfate-utilizing bacteria to flourish, competing with methanogens for the same energy sources which are needed in the formation of methane. Sulfate in wastewater, therefore, tends to lower the reactor performance (Sarner *et al.*, 1988).

Sulfate-reducing bacteria in the wastewater reduce sulfate to sulfide, a very toxic form. However, adverse effects of sulfide in the two reactors during the step-up operation period could be rather insignificant due to the following reasons:

- Dilution certainly helped, especially during the start-up process (Lettinga *et al.*, 1985). The feed was never full-strength. The highest feed strength came near 60 g COD liter<sup>-1</sup>, while the typical strength for raw baker's yeast wastewater used in this study ranged from 70 to 100 g COD liter<sup>-1</sup>.
- The pH's within the reactors were either within or near the optimal range of 7.5 to 8.0. In this pH range, a good part of sulfide dissociates into HS<sup>-</sup>, a form less toxic to bacteria (Sarner *et al.*, 1988).
- The COD/SO<sub>4</sub><sup>=</sup> ratio of the feed has always been above 10 g g<sup>-1</sup>, which means that natural gas stripping of H<sub>2</sub>S was sufficient to keep H<sub>2</sub>S toxicity to a rather insignificant degree (Lettinga *et al.*, 1985).
- The seed sludge had already been acclimated to the wastewater prior to being used to seed the reactors.
- Sizeable concentrations of bacteria have attached to the reactor wall. Reactor B, particularly, enjoyed the added advantage of more attached biomass through the fixed medium. Fixed biomass has been demonstrated to be much more tolerant to stress and toxic compounds (Lo *et al.*, 1984).

## 2) Reactor Profile Study

The chemical analyses conducted on the daily effluent provided information about the reactor as a whole because the effluent was a daily composite. For bench-top laboratory-scale reactors (e.g., 4-liter cylindrical reactors), the above approach is adequate. However, for larger reactors like the two reactors used in this experiment there was the need of knowing the values of parameters being monitored (e.g. pH) at different locations in the reactors in order to better understand how wastewater is being treated in relation to the location in the reactor. In this part of the study, the interest has been focused on sludge concentration, pH, COD, and VFA along the height of the reactors— and thus the name "profile study".

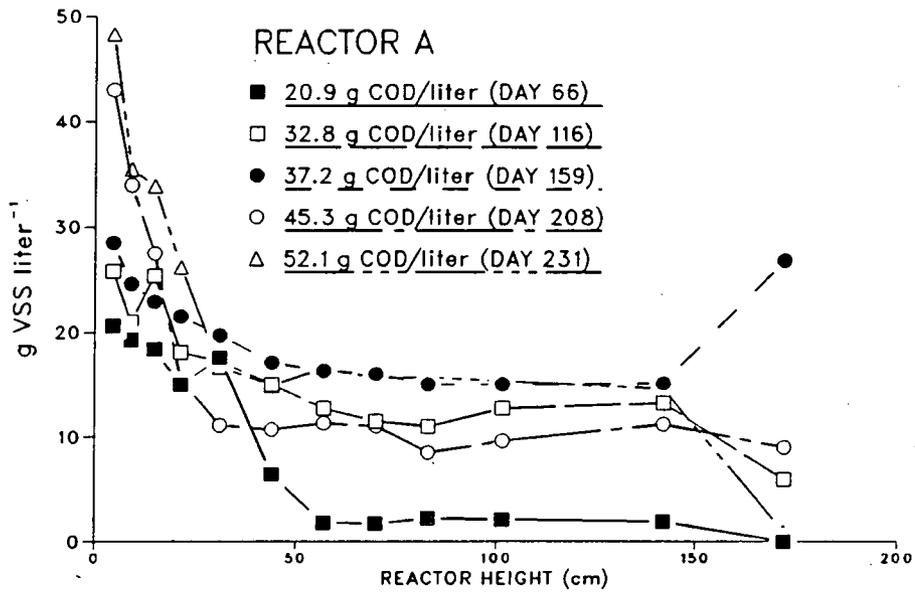
### ***a) Sludge Profile and Total Sludge Growth***

The profile of sludge concentration, expressed in g VSS liter<sup>-1</sup>, along the height of the reactor is presented in Figure 5.4. The general trend shows that the sludge concentration was much higher at the bottom of the reactor, and it tapered off to some extent and levelled off (i.e., VSS concentration became more or less constant) above 50 cm of reactor height, measured from the bottom. The highest point (above 170 cm) corresponded to the sludge sampling port above the three-phase separator. The thick sludge at the bottom was the sludge bed, and the thinner sludge on top of the first was the sludge blanket. The sludge bed, being muddy in this study, tended to hamper smooth fluid flow causing channeling so that feed material preferentially passed through the cracks in the bed. In contrast, the sludge blanket was better mixed and approached complete mixing in the region of constant concentration (Yan *et al.*, 1989b).

The common pattern for both reactors is that, as the organic loading rate increased, the sludge blanket also expanded upwards. First, as biogas was being produced, the sludge bed was lifted upwards by the gas bubbles. As more gas was produced, more flotation was created by the gas bubbles, and thus more sludge was lifted upward. Second, when organic loading became too high, the sludge could not digest the incoming wastewater as quickly as it did at lower organic loading rates. If such a high loading regime was prolonged, the sludge will not be able to maintain its vitality, and its settleability would deteriorate as a result. Poorly settled sludge tended to suspend and/or float to the top of reactor. In this experiment, when poor settleability occurred, reactor treatment efficiency dropped even though the reactors' sludge-retaining ability might have prevented a good percentage of sludge from being washed out.

From the behavior the sludge profile, a record of history of reactor performance could be obtained. At an organic loading rate of 2.6 g COD liter<sup>-1</sup> day<sup>-1</sup>, reactor A has

## SLUDGE PROFILE



## SLUDGE PROFILE

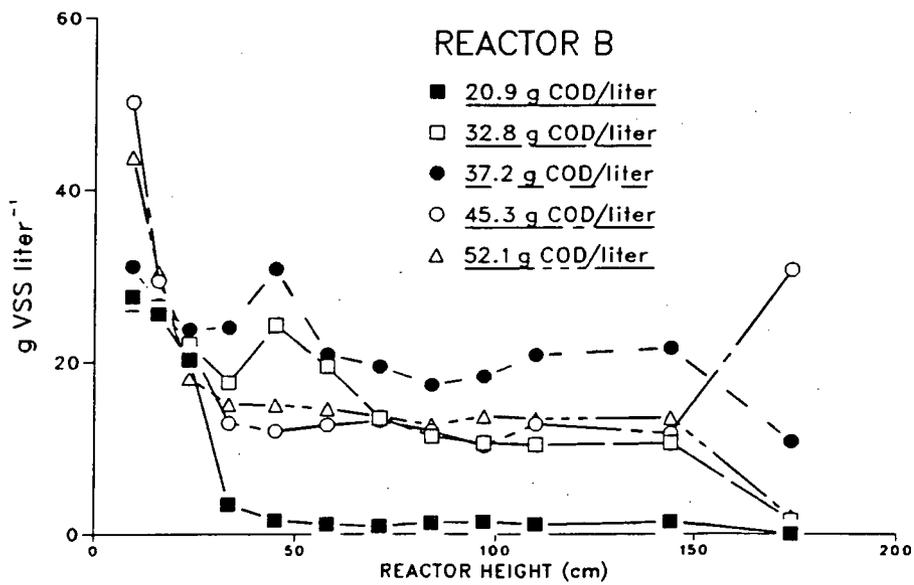


Figure 5.4: Sludge profile versus reactor height

a well-settled sludge bed (Figure 5.4a). Most sludge concentrated in the first 50 cm of reactor height, measured from the bottom; there was very little sludge in the blanket. At a higher loading of  $4.3 \text{ g COD liter}^{-1} \text{ day}^{-1}$  (day 116), the bed expanded considerably so that the blanket concentration rose from about  $2 \text{ g VSS liter}^{-1}$  to  $12 \text{ g VSS liter}^{-1}$ . When the loading was  $4.8 \text{ g COD liter}^{-1} \text{ day}^{-1}$ , the blanket concentration reached its peak of  $20 \text{ g VSS liter}^{-1}$  at day 159 which corresponded to the time when reactor A's treatment was poorest (also compare with Figure 5.2). During the same period, a large amount of floating sludge collected at the top of the reactor, which resulted in a high sludge concentration at the highest sampling port ( $26.8 \text{ g VSS liter}^{-1}$ ). By day 208, the highest bed sludge concentration had risen to  $43.0 \text{ g VSS liter}^{-1}$ , an increase of 38%, and the blanket sludge concentration had slightly lowered. The profile at day 231 was almost identical to that of day 208. One thing common to the last two profiles was that no longer was there excessive floating sludge at the top of reactor. It was felt that the reactor was either adapting to the high loading regime, or enough sludge had been lost so that a new "equilibrium" was reached. Ordinarily, when a reactor is subject to a new higher organic loading rate, its gas production increases and is followed by a surge in sludge washout. If the new operating conditions are well within the capability of the reactor, adaptation and stabilization should be expected. This tendency is best observed in Figure 5.2. As the microorganisms adapted to the new environmental conditions and as they were lost or produced, the total amount of sludge and its distribution within the reactor changed so that at each sampling time was a new equilibrium.

Figure 5.4b shows that, while having a similar overall appearance, reactor B's highest sludge bed concentrations (i.e., concentrations for the lowest sampling port for the five profiles) were generally higher than reactor A's counterparts:  $27.6$  to  $50.2 \text{ g VSS liter}^{-1}$  for reactor B versus  $20.6$  to  $48.3 \text{ g VSS liter}^{-1}$  for reactor A. Another

interesting observation was the "time lag" in reactor B as compared with reactor A. It has been noted that reactor A experienced extensive sludge washout observed in the profile of day 159; however, such sludge washout was not observed in reactor B (indicated by the excessive sludge concentration at the top sampling port) until day 208. It was felt that the fixed-film structure in reactor B must have been responsible for such a delay. Since a fixed-film structure promotes the attachment of bacteria (biomass), one normally thinks that less sludge is freely moving and hence washed out of the system. Similarly, it took longer for reactor B to lose the same amount of sludge—the reactor was said to be more tolerant to loading stress.

The time profile of total sludge, expressed in gram of VSS, in the reactor is shown in Table 5.3. At the beginning of the experiment, each reactor was seeded with 58.3 g VSS. As time went by and the organic loading rate rose, the total amount of sludge in each reactors increased accordingly. There was a steady sludge increase in the two reactors up until day 159 (organic loading rates up to 4.8 and 5.9 g COD liter<sup>-1</sup> day<sup>-1</sup> for reactors A and B respectively). The total sludge figures for this period are in keeping with the observations made in previous sections (Figures 5.1, 5.2, 5.3). The two reactors were under little or no stress. However, after day 159, as the organic loading rate was raised to around 9 g COD liter<sup>-1</sup> day<sup>-1</sup>, signs of stress in both reactors manifested as a decrease in total sludge. Between days 159 and 208, the total sludge decreased for both reactors indicating that sludge washout was higher than growth so that the net growth was negative. It was clear that the bulk of the sludge washout took place during this same period, when the reactors, especially A, were under much loading stress.

**TABLE 5.3: Sludge profile**

Day	Sludge in Reactor		Loading Rate		Sludge Loading	
	g VSS		g COD liter <sup>-1</sup> day <sup>-1</sup>		g COD g <sup>-1</sup> VSS day <sup>-1</sup>	
	A	B	A	B	A	B
1	58.3	58.3				
66	86.6	78.3	2.6	2.8	0.49	0.57
116	215.0	228.1	4.3	5.0	0.32	0.35
159	312.6	379.5	4.8	5.9	0.25	0.25
208	227.3	299.3	9.4	8.3	0.66	0.44
231	293.3	270.3	6.2	6.8	0.34	0.40

Comparing between the reactors, the maximum total sludge of reactor B was always higher than reactor A up until day 231. The higher total sludge in reactor B was one reason why reactor B's methane production was better than that of reactor A. After day 231, the total sludge of reactor B turned out to be lower than that of reactor A. One would expect reactor B to perform less well than reactor A. However, the data presented in Figure 5.2 do not support this rationale. It should be noted that the total sludge parameter does not account for all sludge present in the reactor. In the case of reactor B, it was found that all ropes were covered with thick sludge. Thus, even though the total sludge figure for reactor B at day 231 was less than that of reactor A (270.3 g VSS for A as opposed to 293.3 g VSS for B), the additional sludge attached on the ropes in reactor B probably offset the difference in sludge mass, and was more active and productive than the sludge in reactor A.

There is a relation between the total amount of sludge in the reactor at a given time and the organic loading rate that is imposed on the system. This relation is the sludge loading (or food-to-sludge ratio) and is expressed in g COD g<sup>-1</sup> VSS day<sup>-1</sup>. According to Wu *et al.* (1985), there is an optimum value for sludge loading, 0.6 g COD g<sup>-1</sup> VSS day<sup>-1</sup>, at which the growth conditions are most favorable. Based on this

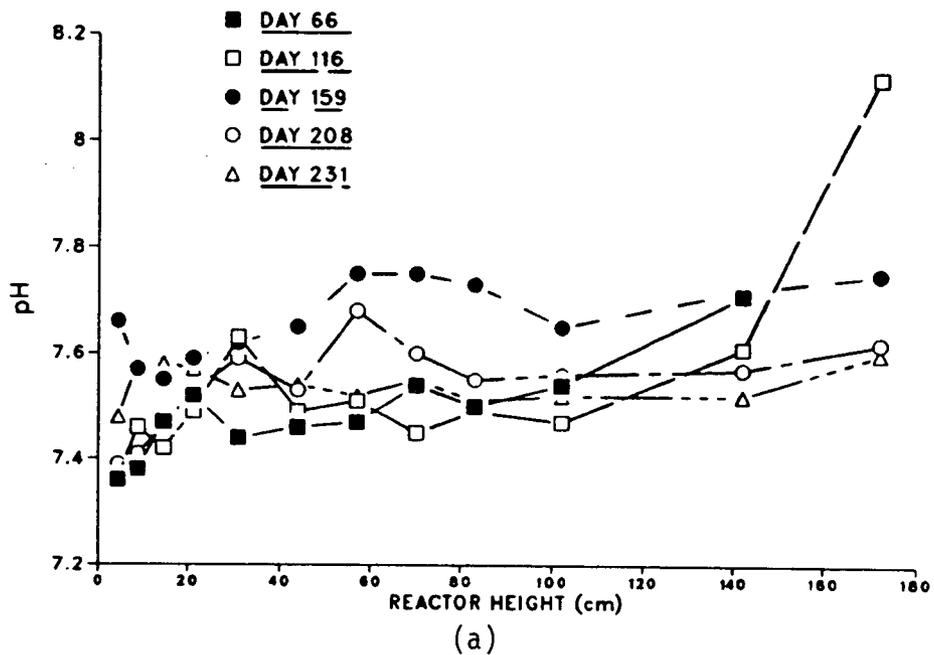
optimum sludge loading, it appears that the two reactors were underfed most of the time during the course of the experiment. Except for the single peak sludge loading rates of 0.66 and 0.57 g COD g<sup>-1</sup> VSS day<sup>-1</sup> for A and B respectively, reactor A's sludge loading rate ranged between 0.25 and 0.49 g COD g<sup>-1</sup> VSS day<sup>-1</sup>, and reactor B's between 0.25 and 0.44 g COD g<sup>-1</sup> VSS day<sup>-1</sup>. However, an examination of Figures 5.1 to 5.3, it does not seem likely that a higher organic loading rate at the very end of the experiment would lead to better methane yields and system performance. Maybe reactor B would give a higher volumetric methane production, because it was not clear whether it would rise or drop. Presumably, if a program of higher sludge loading and, especially, higher hydraulic loading rates (to impose selection pressure) was initiated right from the start of the step-up period, more granular and active sludge would have been developed, leading to better reactor performance (Wu *et al.*, 1985; Lettinga *et al.*, 1985).

### ***b) pH Profile***

The pH along the height of the reactor is depicted in Figure 5.5. It can be seen that the profile pH's of both reactors were all well above 7 and mostly between 7.4 and 7.8. In addition, it is noted that pH generally rose with the height of the reactor; that is, the further up it was from the bottom of the reactor, the higher was the pH. This observation is in keeping with the mechanism of anaerobic digestion. Wastewater is degraded more and more up the reactor; in the same way, the pH increases as VFA's are digested and hydrogen utilized to form methane gas.

Moreover, there was no abrupt increase or decrease in pH. The pH is an indicator of "well being" of the underlying fermentation process. From an oversimplified point of view, anaerobic digestion consists of fatty acid formation and methanogenesis. When there is more fatty acid being formed than it can be converted

### pH PROFILE (Reactor A)



### pH PROFILE (Reactor B)

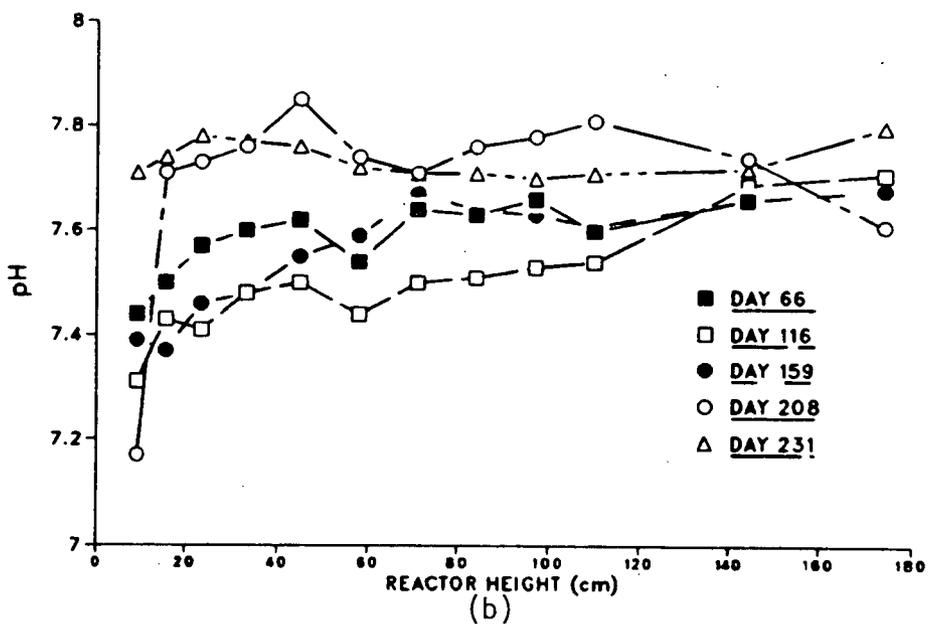


FIGURE 5.5: pH versus Reactor Height

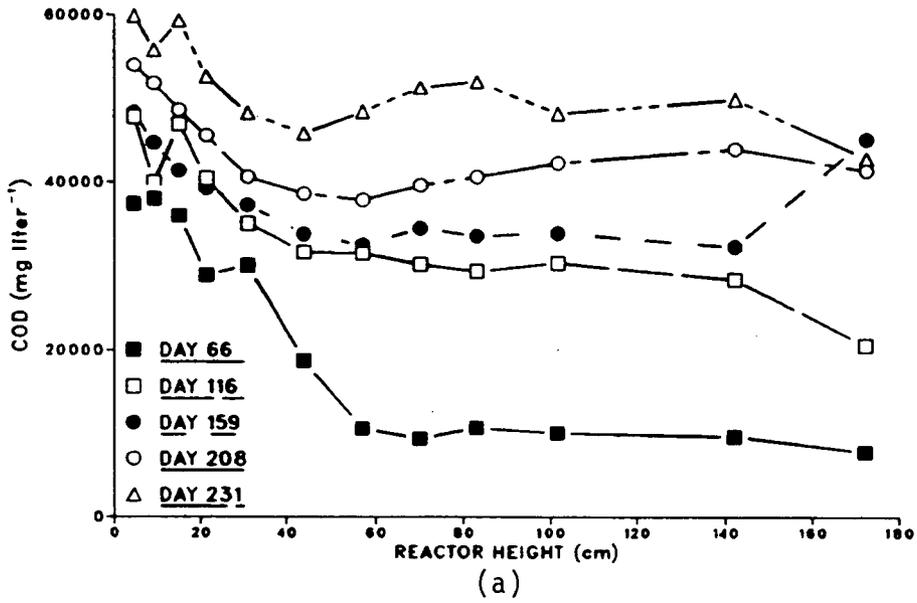
in methane, VFA accumulation occurs and pH drops. Methane production in this case is the limiting stage. The reverse is also true. When methanogenesis proceeds faster than acidogenesis, little fatty acid is left unconverted to methane; pH rises as a result. In the latter case, acid generation is the bottle neck of the fermentation process. Therefore, basing on the foregoing presentation, the absence of pH stratification (i.e., drastically different pH's within a reactor) in the two reactors indicates that acidogenesis was the limiting stage of the whole of fermentation process. This finding is substantiated by the fact that baker's yeast wastewater consists mostly of hard-to-degrade organic matters. In contrast to this observation, Yan *et al.* (1989b), treating cheese whey (readily degradable), reported the presence of two zones in relation to pH, that is, a distinctively lower pH in the acidogenic zone on the bottom of the reactor and a much higher pH in the methanogenic zone above the first zone.

### *c) COD Profile*

The total COD profile based on the five profile samplings is shown in Figure 5.6. There are some important observations to be made. First, in both reactors, the profile curve rises with increasing influent COD concentration. The feed strength always increased in the course of the experiment: from 20 to 52 g COD liter<sup>-1</sup> for the time span of the five profile samplings. This finding is reasonable because, as feed COD concentration rises, the residual COD increases and hence the profile is expected to shift upward (i.e. increase in COD) and may or may not assume a different configuration.

Second, the sludge blanket in each reactor expanded over time. At the time of the first profile sampling (day 66 profile), most of the sludge concentrated on the bottom (the bed) of the reactor leading to high COD concentrations, while the blanket COD concentration was relatively low compared to that of the sludge bed. Sludge

**TOTAL COD PROFILE  
(Reactor A)**



**TOTAL COD PROFILE  
(Reactor B)**

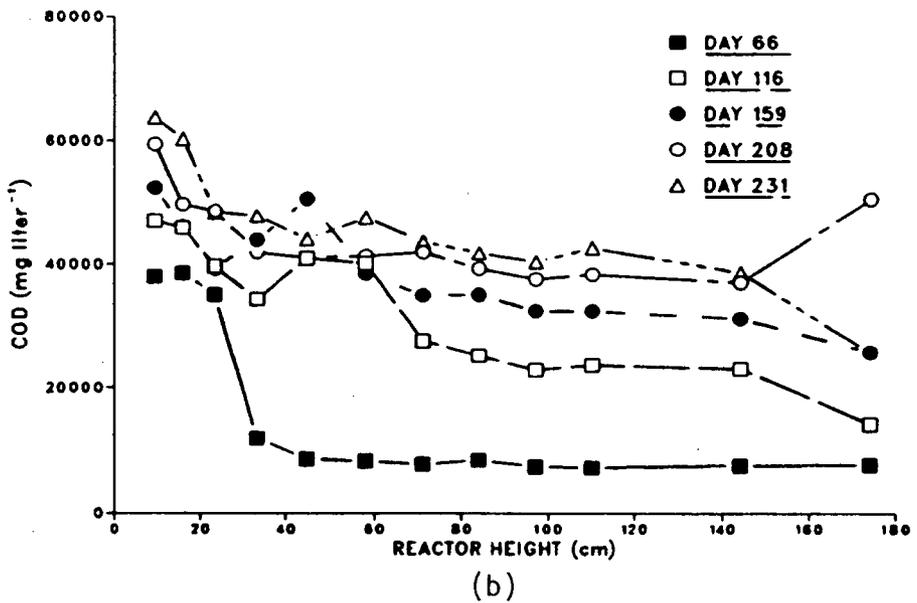
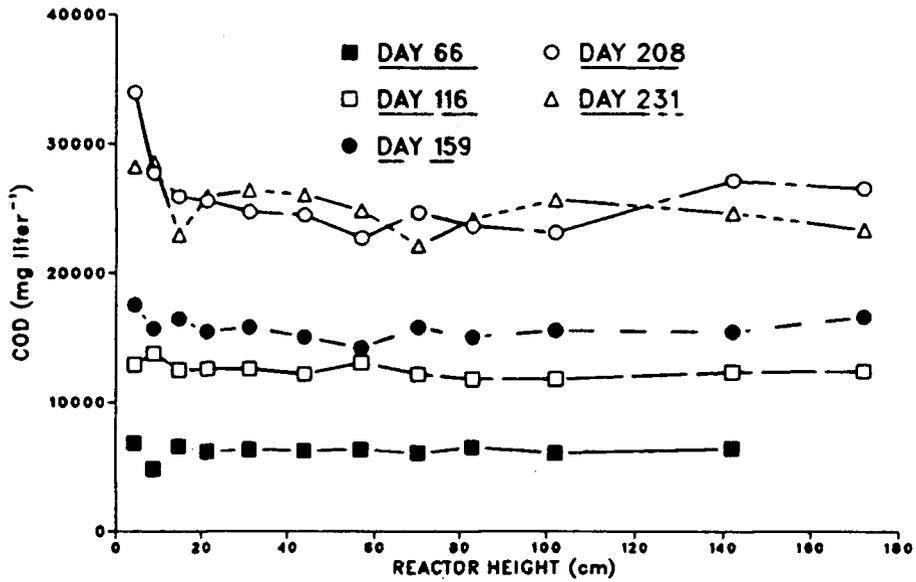


FIGURE 5.6: Total COD versus Reactor Height

settleability at this time was good and sludge activity was high. Over time, however—as buoyancy due to gas bubbles became more significant, and as sludge settleability deteriorated due to increased stress—much of the bed sludge became suspended tended to float to the top of reactor. A direct consequence of all this development is that the difference between bed sludge concentration and that of the blanket became less and less significant. In short, towards the end of the experiment, the entirety of each reactor's column was filled with poorly-settled and, very likely, low-activity sludge. The same conclusion can be drawn from the decreasing difference in sludge concentration along the reactor height (Figure 5.4). Another consequence is excessive sludge washout, which was readily verifiable at and after day 159.

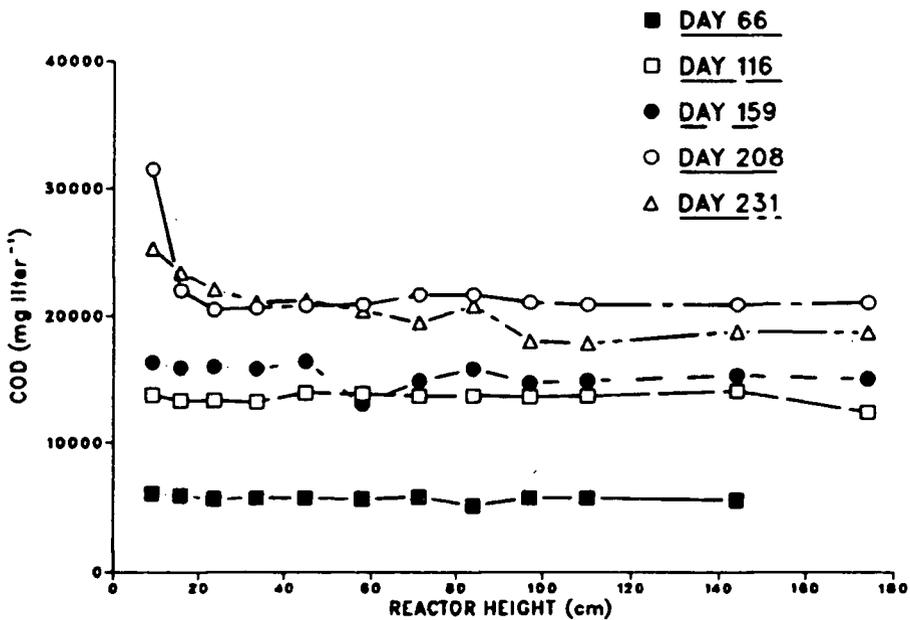
Total COD includes the COD contributed by the sludge and wastewater, so that it can be higher than feed COD alone. This case is especially true in this experiment because COD removal was relatively low and blanket sludge concentration was high. Figure 5.7 (soluble COD profile) helps explain something subtle about the nature of the wastewater being studied that Figure 5.6 does not readily show. It is clear in Figure 5.7 that all soluble COD profiles are essentially horizontal, as opposed to those of total COD in Figure 5.6. This pattern indicates that there was no substantial COD break down 10 cm above the reactor base. In other words, the bulk of the COD break down took place between the bottom of the reactor and the lowest sampling port. This finding underscores the fact that baker's yeast wastewater is hard to degrade. Since dense sludge was at the bottom of the reactor, a lot of digestion took place there. After the wastewater went past the sludge bed, due to its slow rate of biodegradability, it would not undergo visibly significant degradation in terms of soluble COD for the remaining length (or residence time) of the reactor. In terms of total COD, however, it is evident from Figure 5.6 that most of the time the three-phase separator did separate out substantial non-soluble COD (most likely buoyant sludge) from the treated effluent

**SOLUBLE COD PROFILE  
(Reactor A)**



(a)

**SOLUBLE COD PROFILE  
(Reactor B)**



(b)

FIGURE 5.7: Soluble COD versus Reactor Height

overflow (see the total COD numbers at the highest sampling port of the two reactors). It can also be seen that the sludge-retaining capability of the separator became more pronounced as the sludge concentrations increased. The fact that most of digestion took place at the bottom of reactor and that the separator generally was able to remove a significant percentage of non-soluble COD suggests that shortening of the reactor length in the low breakdown region will probably not affect significantly the treatment efficiency of the system.

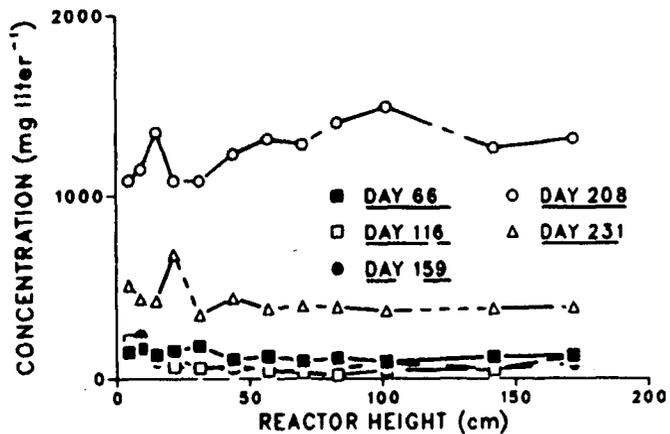
#### ***d) VFA Profile***

Figures 5.8 and 5.9 depict the VFA profiles for the two reactors. In general, even though reactor B's VFA levels were mostly lower than those of reactor A, the first three profiles (those at day 66, 116, and 159) look similar for the two reactors because of the low concentrations. VFA profiles at days 208 and 231 are rather outstanding because of increasing loading stress imposed on the reactors during this period. A common aspect to all VFA graphs is that the profile at day 231 is always lower than that of day 208. It is not hard to figure out the cause of this pattern: the imposed organic loading rate before and at day 231 was lower than the counterpart at day 208.

Another observation is that there seems to be more VFA degradation, especially the profiles at day 208, along the reactor height for reactor B than A. Whereas reactor A's profiles tend to be fairly horizontal for all four VFA's, reactor B's profiles show considerable slope particularly near the bottom of the reactor. More active sludge bed and the fixed-film structure are believed to be responsible for such a difference in reactor B.

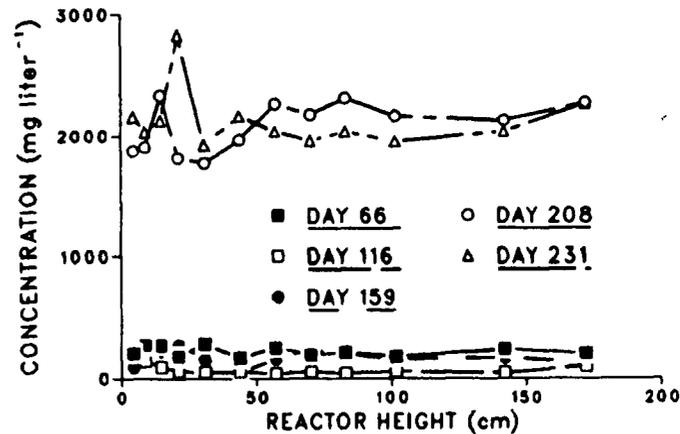
Figures 5.8a and 5.8b shows that reactor A's acetic acid levels are well beyond the suggested failure level of  $800 \text{ mg liter}^{-1}$  (Hill *et al.*, 1988) whereas those of reactor

**ACETIC ACID PROFILE  
(Reactor A)**



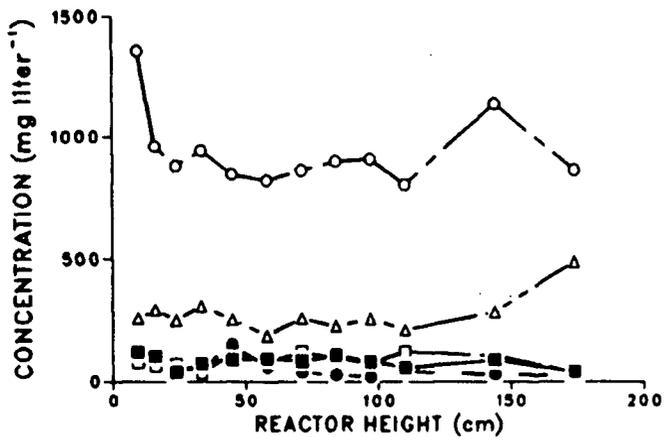
(a)

**PROPIONIC ACID PROFILE  
(Reactor A)**



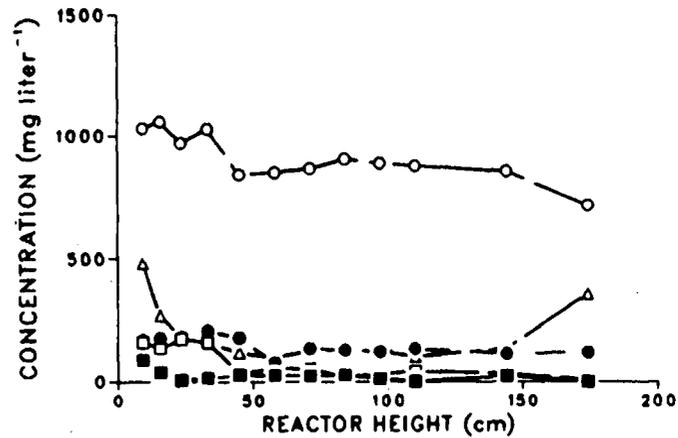
(c)

**ACETIC ACID PROFILE  
(Reactor B)**



(b)

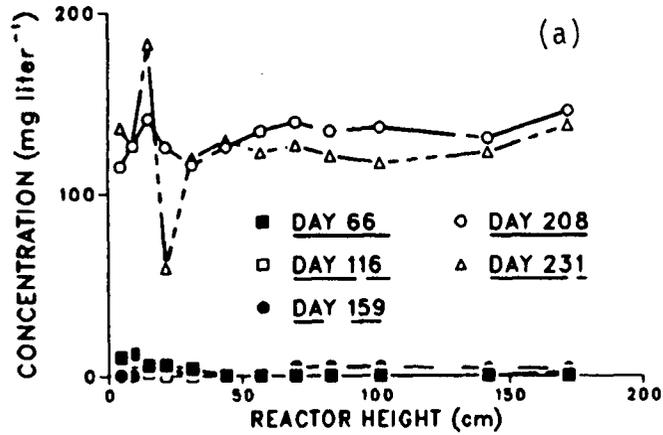
**PROPIONIC ACID PROFILE  
(Reactor B)**



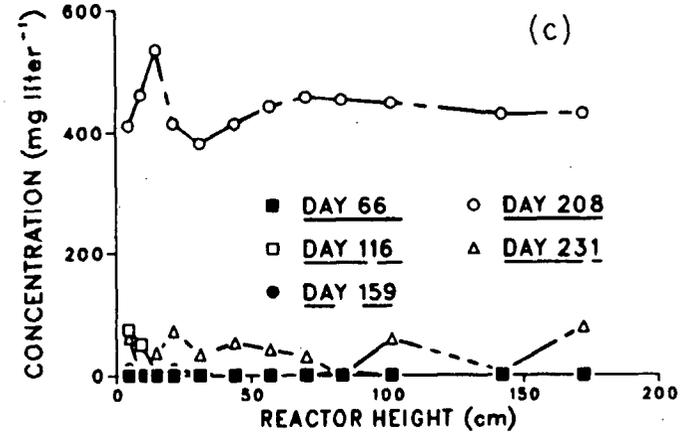
(d)

FIGURE 5.8: Acetic and Propionic Acids versus Reactor Height

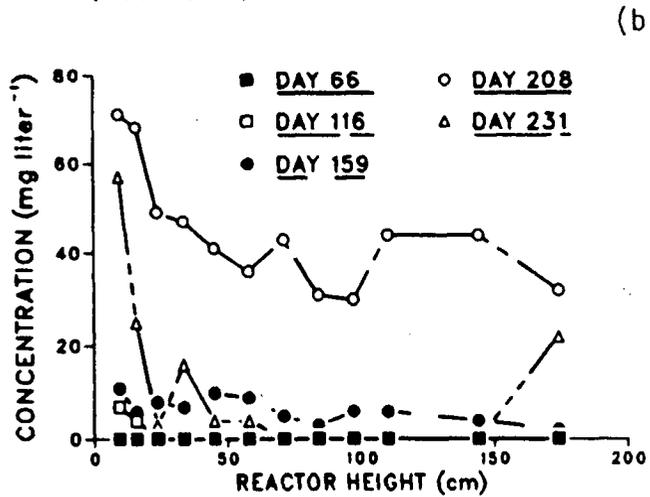
**ISOBUTYRIC ACID PROFILE  
(Reactor A)**



**BUTYRIC ACID PROFILE  
(Reactor A)**



**ISOBUTYRIC ACID PROFILE  
(Reactor B)**



**BUTYRIC ACID PROFILE  
(Reactor B)**

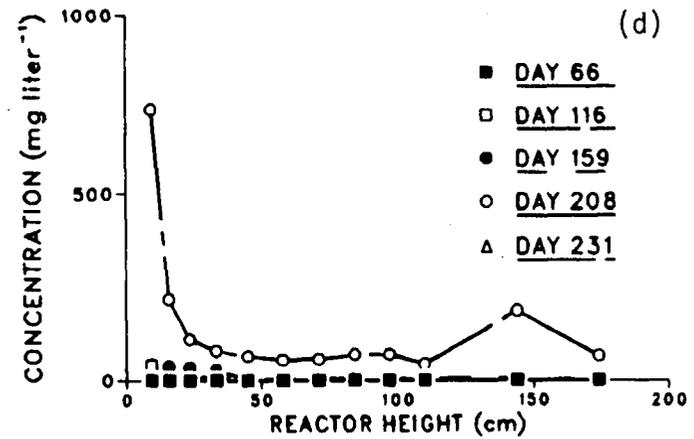


FIGURE 5.9: Iso- and Butyric Acids versus Reactor Height

B are slightly over the failure level. Relatively speaking, reactor B is surviving better than reactor A. These results are found to match with those presented elsewhere in this thesis (under "Step-up Operation").

The propionic acid graph is the key to showing the superiority of reactor B over A (Figures 5.8c and 5.8d). Reactor B's propionic acid levels are drastically lower than those of reactor A— about half of those of reactor A. In contrast, whereas reactor A's propionic acid profile is virtually horizontal and is at around 2000 mg liter<sup>-1</sup>, reactor B's go from 1000 mg liter<sup>-1</sup> at the bottom of the reactor to below 800 mg liter<sup>-1</sup> at the top of reactor. Reactor B is definitely more active than reactor A in digesting propionic acid; the accumulation of VFA's eventually leads to process failure of reactor A (Hill *et al.*, 1988).

Long-chain VFA's are equally important for determining reactor health (Figure 5.9). Hill *et al.* (1988) claimed that iso-butyric acids in excess of 15 mg liter<sup>-1</sup> mean impeding digester failure. Based on this figure, reactor A was definitely in the process of failing (over 100 mg liter<sup>-1</sup>) at and after day 208. In turn, reactor B's iso-butyric acid levels did not exceed the criterion value except at and after day 231. However, it should be noted that reactor B's exiting iso-butyric acid level was about 30 mg liter<sup>-1</sup> and, in addition, that some reduction in VFA usually took place between the top sampling port and the effluent collector. In effect, the implication is that the actual iso-butyric acid concentration should be less than 30 mg liter<sup>-1</sup>. Indeed, a concentration of 20 mg liter<sup>-1</sup>, from Table 5.3 (day 237), would be a reasonable number. In summary, reactor B was on the "border line" in relation to failure.

## B. Chapter Conclusion

- The maximum biogas methane composition was 54% (v/v) during the step-up operation phase. A survey of methane compositions of other authors' at

similar loading rates suggested that the maximum methane composition achievable with baker's yeast wastewater was around 60%.

- Reactor A had achieved its maximum volumetric gas production of 0.91 liter  $\text{CH}_4$  liter<sup>-1</sup> day<sup>-1</sup> at 5.8 g COD liter<sup>-1</sup> day<sup>-1</sup> which was still far from the highest loading rate (9.4 g COD liter<sup>-1</sup> day<sup>-1</sup>) applied in this study. Reactor B, however, did not appear to have reached its highest possible gas production even at the maximum loading applied to it (9.1 g COD liter<sup>-1</sup> day<sup>-1</sup>) since it was not clear whether the methane production curve would drop or continue to rise.
- In terms of methane production, additional biomass clung to the ropes and enhanced reactor B's biogas production, both quantitatively and qualitatively, and at the same time rendered reactor B more tolerant to organic loading rates higher than 6 g COD liter<sup>-1</sup> day<sup>-1</sup>.
- Baker's yeast wastewater is not readily degradable since the highest COD reduction achieved in this study was 71% at an organic loading of 2.3 g COD liter<sup>-1</sup> day<sup>-1</sup> (at a HRT of about 7 days). This number is low compared to that of cheese whey, which is very easily biodegraded. At organic loading rates higher than 6 g COD liter<sup>-1</sup> day<sup>-1</sup> (after day 150), COD reduction fell below 50% for both reactors; single-stage treatment at this efficiency in this loading range was considered rather unattractive.
- Even though baker's yeast wastewater rather different than cheese whey, over the organic loading range of 0.91 to 7.8 g COD liter<sup>-1</sup> day<sup>-1</sup>, the methane yield values of the two reactors was approximately 0.3 liter  $\text{CH}_4$  g<sup>-1</sup> COD day<sup>-1</sup> which was surprisingly similar to the methane yield obtained by Yan *et al.* (1989a) from cheese whey with a UASB (0.30 to 0.34 liter  $\text{CH}_4$  g<sup>-1</sup> COD day<sup>-1</sup>).
- Based on Figures 5.1 to 5.3, especially Figure 5.3, the three stages (with respect to stress and failure) that the two reactors went through can be seen as (1) little or no stress in both reactors for the first 153 days; (2) rapidly increasing stress in both reactors (especially in reactor A) from days 153 to 165; and (3) the setting in of failure in reactor A and reactor B's approaching failure from day 165 to the end of experiment.
- Sulfate effects in the two reactors were found to be not very significant mainly because (1) the pH within the reactors were mostly in the 7.5-8.0 range where sulfate toxicity was minimum; (2) the COD/SO<sub>4</sub><sup>=</sup> ratio of the feed had always

been above  $10 \text{ g g}^{-1}$ , which meant that natural gas stripping of  $\text{H}_2\text{S}$  was sufficient to keep  $\text{H}_2\text{S}$  toxicity to a rather insignificant degree (Lettinga *et al.*, 1985); and (3) the seed sludge had already been acclimated to the wastewater prior to being used to seed the reactors.

- The sludge bed was found to expand with increasing organic loading. This bed expansion manifested in the form of global flattening of the sludge profile curves; that is, the difference in sludge concentration between the sludge bed and the top of reactor diminished over time.
- A time lag in the peak sludge washout was observed for reactor B in relation to reactor A (Figure 5.4). Whereas reactor A had its peak sludge washout in the profile of day 159, reactor B had its equivalent in the profile of day 208. Even though reactor B's total suspended sludge (VSS) was less than that of reactor A, reactor B still performed better. Reactor B's ropes were thought to be responsible for such a difference.
- There was no pH stratification in the reactors. Because baker's yeast wastewater is not readily biodegradable, there was no localization of pH as observed in cheese whey by Yan *et al.* (1989b): the presence of two zones in relation to pH, that is, a distinctively lower pH in the acidogenic zone on the bottom of the reactor and a much higher pH in the methanogenic zone above the first zone.
- Figures 5.6 and especially 5.7 revealed that the bulk of biodegradation took place in the sludge bed (the lower 40 cm of reactor height).
- The profile VFA data (Figures 5.8 and 5.9) were in harmony with those of the Step-up operation VFA (Figure 5.3). The profile of day 159 shows that there was little stress. Thereafter, stress manifested in the form of high VFA profiles.

## VI. CONCLUSIONS

- Methane production and COD reduction from baker's yeast wastewater are lower compared to those of more easily biodegradable wastewaters such as cheese whey.
- It appeared that the maximum achievable methane composition for baker's yeast wastewater was around 60% (v/v).
- At about day 10 and day 14 (during the start-up), reactors B and A respectively experienced the negative-to-positive turnover which signalled that organic removal had prevailed over sludge loss.
- Because baker's yeast wastewater is hard to degrade, there was no stratification in pH (i.e. acidic layer in the bottom and methane zone above the first). As a result of its slow degradability, baker's yeast wastewater has a much higher buffering capacity compared to cheese whey.
- Over the range of feed strengths tried, 25 to 35 g liter<sup>-1</sup> (at an HRT of 7 days) appeared to be the optimum as far as overall efficiency was concerned.
- High levels of sulfate in the wastewater did not significantly affect reactor performance both in the start-up and step-up periods because (1) the seeding sludge had already been acclimated to the wastewater; (2) the COD/SO<sub>4</sub><sup>=</sup> ratio was always above 10 g g<sup>-1</sup>; (3) the pH of the reactor mixed liquor was nearly always in the range of 7.5 and 8.0, where sulfate toxicity was minimum; and (4) dilution of the feed material helped alleviate H<sub>2</sub>S effects.
- It was found that, both in the start-up and step-up operation periods, the two reactors produced methane yields (0.30 to 0.33 liter CH<sub>4</sub> g<sup>-1</sup> COD day<sup>-1</sup>) which were comparable to those of a rather different waste like cheese whey (0.30 to 0.34 liter CH<sub>4</sub> g<sup>-1</sup> COD day<sup>-1</sup>).
- The study revealed (as shown in Figures 5.6 and especially 5.7) that the bulk of biodegradation took place in the sludge bed (the lower 40 cm of reactor height).
- The study found (as shown in Figures 5.3, 5.6, and 5.7) that (1) little or no stress in both reactors for the first 153 days; (2) rapidly increasing stress in both reactors (especially in reactor A) from days 153 to 165; and (3) the setting in of failure in reactor A and reactor B's approaching failure from day 165 to the end of the experiment.

- With the ropes as a fixed-film medium, the hybrid UASB reactor (reactor B) was more tolerant to stress and toxic compounds and consistently performed better than reactor A due to its more active immobilized biomass.

## VII. RECOMMENDATIONS

The following recommendations represent areas which did not receive enough attention during the experiment. Hence the author thinks that anyone doing follow-up work for or repeating the present experiment should take these recommendation in to serious consideration.

Listed below are the recommendations:

- In order to promote granulation for a quicker start-up and superior treatment efficiency, selection pressure (i.e. hydraulic loading regime) must be applied and other pertinent criteria discussed earlier followed. In the case of low hydraulic retention time, intermittent feed and/or effluent recirculation may be necessary.
- Based on the optimum sludge loading of  $0.6 \text{ g COD g}^{-1} \text{ VSS day}^{-1}$ , organic loading rates near or at this level should be experimented to see whether granulation will proceed more quickly than it has been with the present study.
- Where low flow rates are applied, caution must be taken to prevent clogging in the feed line system. Supernatant obtained by natural gravity method should be used to minimize coarse suspended particles.
- In order to ensure the same flow rate in both reactors, separate pumps and speed controllers are a must.

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