ANAEROBIC FILTRATION OF WASTE WATERS ARISING FROM THE PRODUCTION OF BAKERS' YEAST

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ABSTRACT

A study was carried out to assess the effectiveness of the anaerobic filtration process in providing a relatively simple on-site waste pretreatment system for the wastes from the production of bakers' yeast. These are of a high strength and acidic nature. To maintain practical constraints on the project a real waste water was used. The waste water is low in suspended material, has a high organic matter concentration giving a COD of 70000 to 90000 mg/ℓ (BOD_5 14250 mg/ℓ) and a pH of 4.5 to 5.0. Two experimental anaerobic filter units were constructed of glass cylinders packed with glass 'Raschig' rings as the inert support matrix, and used in the trials. A statistically designed experimental programme was used to test the effects of inflent substrate concentration, hydraulic retention time, temperature and filter unit on the filter response variables. Substrate concentrations of 5500 to 47200 mg COD/ℓ (1000 to 9000mg BOD_5/ℓ) and hydraulic retention times of 1 to 5 days were used which gave rise to organic loading rates of 1.15 to 47.2 kg COD/m^3d (0.2 to 9.0 kg BOD_5/m^3d). Empirical models were derived from the response data,using multiple regression techniques, and describe COD removal rate, total gas production, methane production and conversion of COD to methane in terms of the independent process variables. Results showed that the anaerobic filter achieved COD removals of 34 to 65 percent with corresponding conversions of 24 to 78 percent efficiency to methane at loadings of up to 47.2 kg COD/m^3d. The empirical equations were able to explain 92 to 97 percent of the observed variance. The system was stable over the entire range of applied conditions.

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PREAMBLE

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The work described in this thesis was an attempt to cover the areas of both anaerobic waste treatment and experimental design. The Biotechnology Department at Massey University has always had a strong involvement in the field of industrial waste treatment, dealing principally with liquid waste streams. In addition, the use of statistically designed experimental programmes and the use of mutiple regression techniques for the data analysis was also finding favour increasingly. The problems of a local industry producing a liquid waste of high organic strength and an acidic nature was also an element in the determination of the final scope of the project.

The nature of the waste stream indicated that anaerobic biological treatment was a possible option and in particular anaerobic filtration. The inherent simplicity of the anaerobic filtration process lends itself well to the requirements of an on-site waste treatment plant. The use of a real waste water with its variability was also desired for the study in comparison with the use of a synthetic waste, so that real constraints would be placed on the operating conditions used in the experiments and the results would be directly applicable to the real situation.

The use of a statistical experimental design over a long period of time was required in this case in order to gather data on the effects of process variables on the process which required long periods of time to settle at steady-state conditions after changes in the inputs. The use of a statistical base was necessary so that the regression analysis was simplified.

The empirical equations generated were seen as an alternative

to the attempts to apply equations derived from chemical kinetics to a biological system being operated over a wide range of conditions and using gross measures of input parameters, such as COD for substrate concentration.

The following dissertation covers a discussion of the literature dealing with work carried out on the treatment of yeast plant effluents, the anaerobic digestion process in general and anaerobic filtration, followed by a description of work carried out by the author using the anaerobic filter to treat the waste water. Empirical equations are fitted to the results to describe several system responses and a brief economic analysis of the use of the filter as an on-site pretreatment process is included.

CHAPTER TWO

WASTE WATERS ARISING FROM THE PRODUCTION OF BAKERS' YEAST AND THEIR TREATMENT

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WASTE WATERS ARISING FROM THE PRODUCTION OF BAKERS' YEAST AND THEIR TREATMENT

2.1 Production of Bakers' Yeast

Bakers' yeast is produced commercially to meet the demand for the bread and other leavened products consumed daily by millions of people the world over. The yeast produced is required in a proportion of one to four percent on a moist weight basis of the bakery products, hence large quantities are required each and every day. The industry itself is an old and established one and recent improvements have been restricted to changes in engineering detail and application of the latest advances in biochemistry and genetics as related to the yeast itself.

In all commercial processes, the yeasts reproduce asexually and such propagation consists of the conversion of convenient sources of carbon, hydrogen and oxygen and supplementary nutrients into individual yeast cells. The type of yeast produced must satisfy many consumer requirements, however such considerations are outside the scope of this study. This review will confine itself to the physical and biochemical aspects of bakers' yeast production insofar as it affects the composition of the waste waters.

2.1.1 Biochemistry of Yeast Propagation

The yeasts *Saccharomyces cerevisieae* and *Saccharomyces uvarum* may be grown anaerobically, fully aerobically or at any intermediate oxygenation degree. In the presence of

free oxygen a relatively simple reaction occurs when glucose and ammonia are used as the carbohydrate and nutrient sources. Equation 2.1 describes this aerobic reaction. Under these conditions it may be seen that there are no appreciable quantities of by-product formed except for carbon dioxide

$$\underset{100.0g}{0.55556\ C_6H_{12}O_6} + \underset{5.10g}{0.30017\ NH_3} + \underset{46.63g}{1.45729\ O_2} \rightarrow \underset{43.34g\ yeast}{0.30017\ C_6H_{10}O_3N} + \underset{41.08g}{2.28276\ H_2O} + \underset{67.42g}{1.53234\ CO_2} \quad (2.1)$$

(Harrison, 1971)

and water. Conversely under anaerobic or conditions of limited available oxygen the following reaction (equation 2.2) has been shown experimentally to occur (Harrison, 1971).

$$\underset{100.0g}{0.55556\ C_6H_{12}O_6} + \underset{0.21g}{0.01214\ NH_3} \rightarrow \underset{1.748g\ yeast}{0.01214\ C_6H_{10}O_3N} + \underset{5.241g\ glycerol}{0.05697\ C_3H_8O_3} + \underset{1.42g\ succinic\ acid}{0.0204\ C_4H_6O_4} + \underset{0.05g\ butanol}{0.00071\ C_4H_{10}O} + \underset{0.21g}{0.01164\ H_2O} + \underset{43.32g}{1.03076\ CO_2} + \underset{46.17g\ ethanol}{1.0038\ C_2H_6O} \quad (2.2)$$

From the above equation one notes the large decrease in yeast material produced and increased by-product formation compared to the aerobic phase described by equation 2.1. Principally ethanol is produced during this anaerobic phase. In the simplified equation, succinic acid and butanol are generalised representations of all the organic acids and alcohols respectively and glycerol is the other major product besides ethanol.

By supplying air at rates which provide oxygen conditions between the extremes described by equations 2.1 and 2.2,

both ethanol and yeast mass may be produced in any required proportion - the so called 'Vienna process' (Rose, 1961). Such systems can be controlled by feed-back mechanisms using alcohol and oxygen measurements.

In production of bakers' yeast alone a fully aerobic state must be maintained to produce maximum yields of yeast mass (equation 2.1). To this end the reverse Pasteur effect or Crabtree effect must be avoided by regulation of the assimilable sugar concentration. The Crabtree effect is a phenomenon of yeast metabolism whereby at high sugar concentration the anaerobic metabolism is manifest even in the presence of excess free oxygen. Similarly an induction of a partially fermentative state may be effected by an excessively high yeast concentration which reduces oxygen permeation and increases carbon dioxide concentration. Therefore a very vigorous air flow is required, possibly assisted by mechanical aeration.

On a molar basis, yeast matter has an approximate composition of

$$C_6H_{10}O_3NK_{0.01}P_{0.06}S_{0.03}$$ (Harrison, 1971)

These elements are provided from the sources outlined below. During propagation, a small seed culture of yeast is built up to the final size by successively transferring the culture to larger propagation vessels with the addition of growth medium. A five stage build up of cell numbers is typical. As the yeast cells grow and reproduce asexually, essential elements are taken from the source materials, present in the propagation medium, and incorporated into new cell matter by the biochemical processes inherent in yeast metabolism.

Carbon, hydrogen and some of the oxygen required for yeast production are usually provided in the form of a carbohydrate and cane molasses is frequently used. The molasses,

Table 2.1: Elemental Composition of Commercial Yeasts and Molasses (Harrison, 1971).

Values in kg/100kg of dry matter.

Element	Yeast	Molasses	Minimum Difference
C	45-47	39-41	-4
H	6-6.5	6.3-6.8	-0.2
O	31-32	47-50	+19
N	7.5-9.0	0.1-2.8	-4.7
K	.9-3.5	.8-5.2	+4.3
P	1.1-2.0	.01-.9	-0.2
S	.3-.5	.15-.2	-0.1
Mg	.15-.5	.007-.75	+0.6
Ca	.04-.9	.018-1.2	+1.16
Na	.02-.2	.82-1.4	+1.38
Zn	.004-1.3	.0006-.013	+.009
Fe	.003-.1	.01-.021	+0.018
Cu	.002-.012	.0001-.006	+0.004
Mn	.0004-.0035	.001-.004	+.0036
Co	.0005	.00004-.0001	-.0004
Cl	.004-.1	.13	+0.126
Mo	$5\text{-}9\times10^{-6}$	$9\text{-}26\times10^{-6}$	$+21\times10^{-6}$
I	$5\text{-}40\times10^{-6}$	-	$-\ 5\times10^{-6}$
Pb	.0001-.0007	.0006	+.0005
As	.00001	-	-.0001
Si	-	.028	+.028
Sr	-	.005	+.005
B	-	.0002-.0004	+.0004

a by-product of the sugar refining industry, generally contains about fifty percent assimilable sugars along with other organic compounds, minerals and 16 to 24 percent water. Other carbohydrate sources used commercially are spent sulphite liquor from wood pulping and wood waste itself. Table 2.1 lists the elemental composition of yeast cells alongside that of cane molasses. Since the commercial propagation would seek maximum utility of its carbohydrate source it is apparent that a carbon limited growth medium based on molasses would be principally deficient in nitrogen and phosphorus and to a lesser extent in magnesium, calcium, zinc, iron, copper and other elements found as traces in yeast matter.

The nitrogen sources used to make up the deficit from molasses are added as ammonium salts (principally, ammonium sulphate) and as urea, although this latter is less well utilised by the yeast but is cheaper than ammonium sulphate.

Metabolic phosphorus is provided as phosphoric acid and/or phosphates such as di-ammonium hydrogen phosphate. Elemental oxygen is required for the aerobic propagation of the yeast and this is provided by sparging the fermenter vessel with air.

In addition to the above, large quantities of process waters are required to make up the required volumes of growth medium as well as materials for pH control and anti-foaming.

2.1.2 Process Description

Actual process variables and equipment will differ from plant to plant, but typically a multi-stage propagation with four or five stages is used. Figure 2.1 is a flow diagram for such a process using molasses as the principal carbohydrate source. The propagation tanks Y_i to Y_n are batch fermenters used to build up the yeast numbers to the required level for introduction into the final fermenter volume (V). This final vessel is a batch process with a

continuous feed of nutrient medium. To limit yeast growth and avoid oxygen starvation the nutrient availability is controlled. Initially nutrient levels are kept low, then as cells reach the exponential growth phase they are added to keep pace with yeast growth. Thus at any time there is only a small excess of nutrient over cells available to utilise it.

The operating temperature of the yeast propagation process is $30^{o}C$. The aerobic conversion of glucose to yeast material is a highly exothermic one and considerable amounts of heat must be dissipated from the fermentation vessel to maintain the operating temperature. Cooling coils within the vessels or external cooling jackets are used for this purpose. The free energy change for the reaction has been calculated as approximately 1.55×10^{7} kJ/tonne yeast formed (Harrison, 1971).

Molasses is clarified by centrifugation prior to its pasteurisation and use, to remove sludge which may be toxic to the later fermentation. It is then diluted to the required sugar concentration and becomes the wort used to feed the fermenter. Other inputs to the fermenter, are the nitrogen source, mineral preparations and compressed air. Because of the low solubility of oxygen in the growth medium and air contains only 21 percent oxygen, about 40 times the absolute requirement must be supplied. This amount of air is equivalent to 10^{4} m^{3} per tonne of commercial yeast produced.

Specific growth rates of yeast under favourable conditions are as high as 0.6 h^{-1}, this coupled with a yield of 0.43 for conversion of glucose to cell matter gives a glucose utilisation of 1.39 kg glucose/(kg yeast.h) during unrestricted growth and an oxygen utilisation rate of 0.647 kgO_{2}/(kg yeast.h).

2.1.3 Sources of Effluent from Bakers' Yeast Propagation and their Characteristics

The principal effluents arising from the production of bakers' yeast are exhaust air and the various liquid wastes streams indicated in figure 2.1.

Air exhausted from the fermentation tanks is vented to the atmosphere in very large quantities as previously noted and represents no hazard to the environment apart from the characteristic molasses odour. The air from drying the filtered, compressed yeast to active dry yeast, if such a processing stage is included, must be filtered to avoid pollution by yeast dust.

The principal waste from a bakers' yeast plant which poses a pollutional problem is, therefore, liquid in nature. These liquid wastes are the molasses sludge from the clarifier, spent fermentation liquor, yeast cream wash water, filter press water and fermenter cooling water.

The fraction of the total pollutional load to each of the streams is given in Table 2.2, from which it may be seen that the majority of the oxygen demand of the yeast plant effluent arises from the spent fermentation media.

TABLE 2.2 : Pollutional Load of Liquid Waste Streams (Stander *et al*, 1971)

Stream	% of total volume	% total pollution*
Molasses sludge	negligible	15
Spent nutrient	20	70
Cooling water Wash water Filter press liquor	80	15

* 5 day biochemical oxygen demand (BOD_5) basis

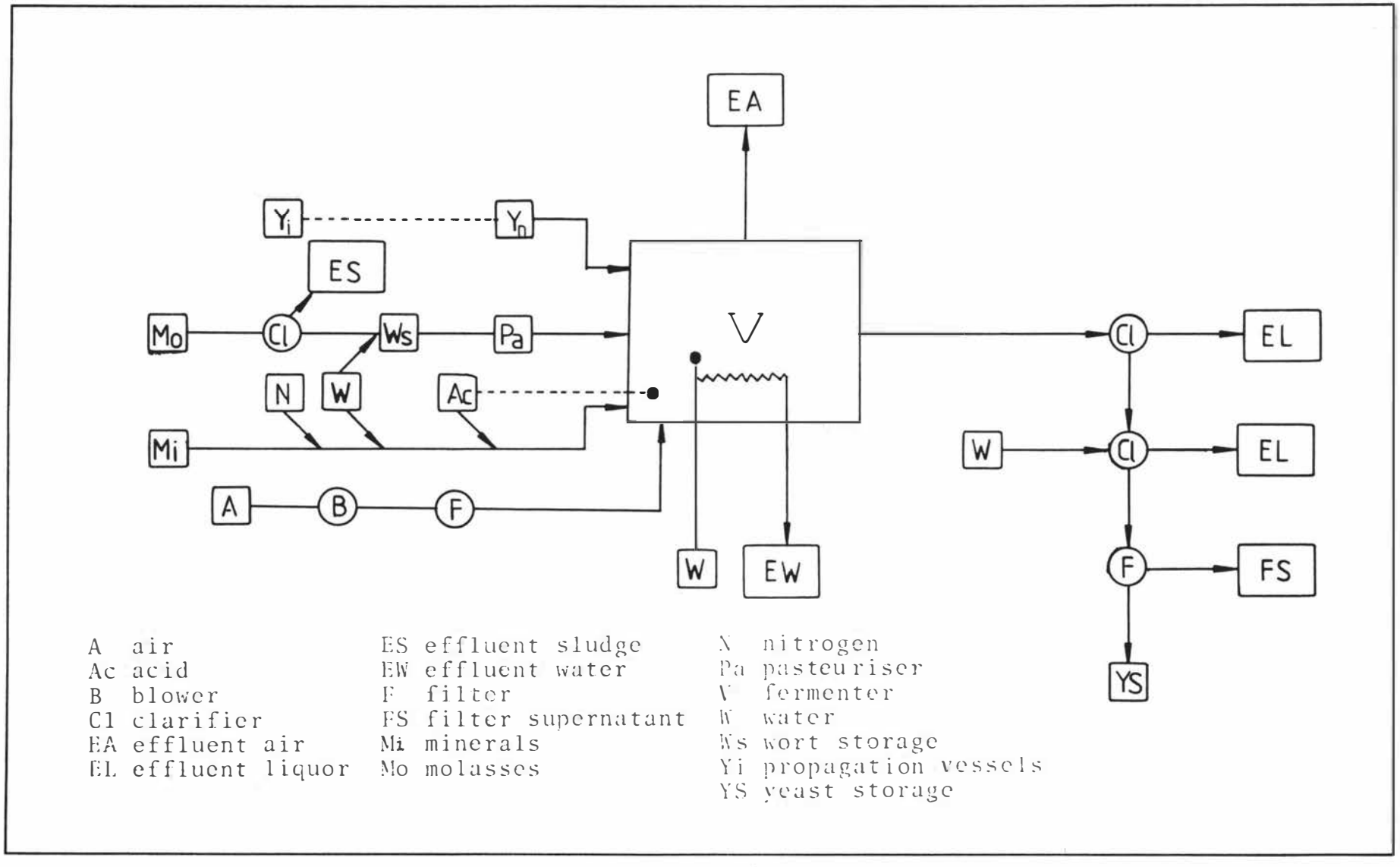


FIGURE 2.1: Flow diagram for the production of commercial bakers' yeast from molasses. (Based on Harrison,1971)

The molasses sludge is readily separated from the other waste streams as it leaves the centrifugal clarifier as a compact sludge.

The spent fermentation medium which comprises only 20 percent of the total liquid waste volume has 70 percent of the pollutional load of the yeast plant associated with it. After propagation of the yeast mass, the BOD_5 of this stream is very high (Table 2.3) due to the presence of the by-products of yeast growth and residual substrate not utilised during yeast growth. The yeast is separated as a cream from the spent medium by centrifugal separators, after which the cream is washed one or more times with fresh water to remove further medium. Table 2.3 lists the important waste treatment characteristics of the spent liquor. Since a batch propagation process is used for commercial yeast production, the spent nutrient media waste stream is not a continuous flow from the plant but occurs also in batches.

TABLE 2.3 : Composition of Spent Liquor from Bakers yeast Propagation
(Trubnick & Rudolfs, 1948)

BOD_5	2000-15000 mg/l
Total solids	10000-20000 mg/l
Suspended solids	50-200 mg/l
Total nitrogen	800-900 mg/l
Sulphate	2000-2500 (as SO_4) mg/l
pH	4.5-6.5

The important points are the high BOD_5 and total solids of the waste stream and the low suspended solids levels. This low concentration of suspended solids is due to the yeast cream separation process. The waste is acidic due to the pH requirement for yeast growth in the range 4.0-5.5 (Carr, 1970).

The other liquid wastes from yeast production comprise large volumes of low BOD_5 waters.

Many yeast plants are located within population centres and disposal of the waste waters poses serious problems.

Local and national regulations often forbid the disposal of any untreated waste water into a receiving water. In New Zealand the disposal criterion is based on maintaining receiving water standards and therefore the quantity of BOD_5 and suspended material discharged per unit time is regulated. A local authority would be reluctant to accept yeast plant effluent for disposal via its municipal sewerage system because of the effluent's large amount of organic material passed on to the receiving water. In addition, the acidic nature of the yeast effluent and its batch discharge nature pose problems for downstream treatment facilities. If such facilities incorporate biological treatment stages, several weekly slug doses of a high BOD_5 acidic waste could easily overload them.

In most cases if a local authority does accept industrial wastes for treatment and disposal, a levy is imposed on the industry based on the volume and strength of the discharge.

As most bakers' yeast plants are sited within municipal boundaries such levies on waste discharge are particularly important since the economic viability of bakers' yeast production can be largely dependent on the costs of waste treatment (Peppler, 1970).

2.2 Treatment of Waste Waters Arrising from the Production of Commercial Bakers' Yeast

Waste waters from a yeast plant have been shown to comprise the spent fermentation media, yeast cream wash waters, filter press water and fermentor cooling water. The latter two items have a minimal effect on the pollutional load from the yeast plant but greatly affect the overall volume

of the waste water and thereby the concentration of the mixed wastes. A suggested practice (Stander *et al*,1971) is to keep these relatively cleaner waters separate from the spent nutrient, yeast cream washes and the sludge from molasses clarification . The molasses sludge may be confined as a compact mass and disposed of separately by, for instance landfill, thus removing fifteen percent of the plant's organic waste. The bulk of the remaining BOD_5 is then concentrated in a relatively small volume; which may be treated on site, mixed, after treatment, with the dilute wash and cooling waters and discharged.

The treatment and disposal of the spent nutrient and yeast cream wash waters is the basis of studies on yeast plant effluent treatment; and in this study, except where noted, yeast plant effluent refers to these waste waters.

Yeast plant effluent is characterised by the following points: (a) a pronounced brown colour, (b) a high concentration of organic material in a true solution or colloidal state, (c) a tendency toward anaerobic acid fermentation, (d) the formation of hydrogen sulphide by reduction of sulphates and/or decomposition of proteins. (Londong, 1968)

Up until about 1948, the bulk of information on treatment of yeast plant effluents had been obtained from data on industrial discharges from brewing and fermentation industries. The effluents from these industries are similar in nature to the yeast effluent (Trubnick & Rudolfs, 1948). To some extent this is still the case and in the following review some reference to industrial discharges similar in nature to yeast plant effluent will be made where relevant.

2.2.1 Chemical Treatment of Yeast Plant Effluent

Chemical treatments, which are most often associated with the tertiary or polishing stage of waste treatment

technology, have found little application in the treatment of yeast plant effluent. Early studies reported BOD_5 removals of zero to 15 percent with an average of 10 percent for an unspecified chemical treatment and 4 to 40 percent with an average of 28 percent BOD_5 removal for electrodialysis (Trubnick & Rudolfs, 1948).

Chemical coagulation trials have found that excessively high doses of coagulent were required to remove minimal amounts of organic material. Lime dosing at 5000 mg/ℓ removed 20 percent of the COD, coal and smokeless fuel as carbon adsorbents were only marginally effective (Stander *et al*, 1971).

Some trials have been carried out to assess the efficacy of chemical coagulents as colour removal agents in yeast plant effluent treatment. The yeast waste previously treated by activated sludge could have some residual COD and colour removed by alum and ferric chloride ($FeCl_3$) coagulation. The addition of 4000 mg/ℓ of alum removed 28.7 percent of the COD along with 29.9 percent of the colour and $FeCl_3$ removed 22 percent COD and 15 percent colour at the same dosage. Overall colour removal from a two stage activated sludge process followed by 4000 mg/ℓ alum treatment was 84.9 percent (Wu & Kao, 1976).

2.2.2 Aerobic Biological Treatment of Yeast Plant Effluent

Three types of aerobic biological treatment systems have been applied to yeast plant effluent treatment. These are; activated sludge, trickling filters and rotating biological contactors.

All aerobic treatments involve the contact of the waste stream with an active mixed biomass in the presence of oxygen. In activated sludge treatment the biomass is present as a biological sludge admixed with the waste stream to form a homogenous mixed liquor. Oxygen is supplied by sparging

the mixed liquor vessel with air or using surface aerators. Both methods usually also supply the mixing power to maintain the mixed nature of the treatment vessel contents.

In trickling filters, the biomass is present as a film attached to a support medium. The waste stream is distributed over the biomass and oxygen is transferred into the liquid as it passes through the filter voids by natural convection of air within the filter.

Rotating biological contactors consist of a number of discs arranged on a common drive axle. The lower portion of the discs is submerged in the waste stream and a biomass is built upon the discs as they rotate and are alternatively immersed in the waste water and the atmosphere.

In aerobic treatment of waste streams the active biomass utilises organic material in the waste for growth and reproduction of new biomass. In the treatment of some industrial wastes, nutrients may need to be added if the waste is deficient in them.

In aerobic biological treatment, the oxidation of organic matter to carbon dioxide and water releases a considerable amount of energy which is available to the biomass for growth. Hence only about 50 percent of the organic matter removed from a waste stream is actually stabilised, the remainder being converted from a soluble or suspended form into an insoluble biological sludge which must be disposed of separately.

Typical operating parameters for the aerobic systems mentioned above are: feed concentrations of 50 to 4000 mg BOD_5/ℓ and retention times varying from 0.5 to 24 h depending on the system configuration (Grady & Lim, 1980). The use of pure oxygen rather than atmospheric air has been investigated for these aerobic processes and has found favour in some applications.

Activated Sludge

A summary of the results of activated sludge treatment of yeast effluent is presented in Table 2.4. The data in the literature provides an incomplete picture of organic material removal efficiency by the process. However, BOD_5 removals in excess of 80 percent have been generally found although at relatively lower substrate concentrations than the yeast plant effluent has characteristically. The 78 to 96 percent BOD_5 removals reported by Wu & Kao (1976) were for yeast waste diluted with tap water at a ratio of 1:1, however another study (Dazai *et al*, 1966) observed that no improvement was gained by dilution of the effluent prior to activated sludge treatment.

The loading rates cited are higher than the range generally considered desirable for conventional activated sludge treatment.

BOD_5 removal efficiency was found to be independant of feed BOD_5 concentration at feed BOD_5 levels of less than 200 mg/ℓ (Grishna & Klimenko, 1972), although the removal efficiency was dependant on the activated sludge concentration. The BOD_5 removal rate was enhanced at higher operating temperatures but acclimation temperature was relatively unimportant. (Takiguchi, 1967, 1968) The addition of supplementary nutrients to the activated sludge did not improve treatment efficiency in one study; and an absence of protozoa in the sludge was thought to be due to some toxicity other than sulphide (Stander *et al*, 1971). Conversely the addition of urea as a supplementary nitrogen source has been shown to improve both sludge settleability and BOD_5 removal efficiency (Takiguchi, 1967, 1968).

No actual economic analysis of the treatment process of yeast plant effluent by activated sludge was shown for any of the investigators, however Stander *et al* (1971) concluded that, at the retention times evaluated, the high cost of installation and operation of an activated sludge

TABLE 2.4: Activated Sludge Treatment of Yeast Plant Effluent

Feed Concentration BOD_5 mg/ℓ	Feed Concentration COD mg/ℓ	Hydraulic retention time h	Temperature ^oC	Loading rate $kg/m^3.d$	Removal Efficiencies BOD_5 %	Removal Efficiencies COD %	Source
16300	21120			<3	96-98	64-65	Dazai *et al* (1966)
		20			>80		Takiguchi (1967, 1968)
				1.0-2.3	80		1) Londong (1968)
				1.0-6.2	80		2) Londong (1968)
4200					99.6	82-87	Grishna & Klimenko (1972)
		120		6.6		65-70	2) Stander *et al* (1971)
		72		2.9		35-40	3) Stander *et al* (1971)
2350	7500	24	25		78-93	80-85*	4) Wu & Kao (1976)
2350	7500	8+16	25		88-96	96-99*	5) Wu & Kao (1976)

* suspended solids removal

NOTES :
1. Combined yeast effluent and domestic sewage
2. Yeast plant effluent alone
3. Activated sludge treatment of effluent from an anaerobic digester treating yeast effluent
4. Single stage 24h retention time
5. Two stage 8h + 16h retention time

plant could not compete against an anaerobic digestion treatment alternative. They also pointed out that if possible, the yeast plant effluent should be treated in combination with domestic sewage.

This latter alternative is described in detail by Londong (1968). The waste water from a West German yeast plant producing both ethanol and yeast material from molasses could not be treated on site due to technical and economic difficulties and so is treated in association with domestic sewage from a nearby city. The effluent stream was carried 21 km by a pipeline to the treatment facility. Transit took on average one day and the effluent was cooled and the pipline fitted with pressure release valves to reduce the chances of gas production in the pipelines.

After separate pre-aeration stages the domestic sewage and yeast plant effluent are mixed into an activated sludge basin where 80 percent of the common BOD_5 was removed.

In anticipation of removal of the yeast plant to a new location where waste treatment cost was lower, further trials were carried out treating the yeast plant effluent in combination with other industrial and domestic waste waters. The costs of the processes were compared with treatment of the yeast plant effluent on its own. Power consumption for combined treatment was calculated to be 0.85-1.25 kWh/kg BOD_5 removed and 0.3-0.94 kWh/kg BOD_5 removed for separate treatment. The overall cost of aerobic treatment was calculated to be 4-10 percent of the production cost of the yeast.

Trickling Filter

The application of trickling filters to the treatment of yeast plant effluent was reported by Trubnick and Rudolfs (1948) to be able to remove 51 to 87 percent of the applied BOD_5, with an average value of 72 percent. However no data for types of filter or operating parameters were quoted.

More recently a pilot-scale study (Lines, 1968 and Jackson and Lines, 1970) showed that stone-filled biological filters loaded at 0.43 kg BOD_5/m^3d could achieve an 80 percent BOD_5 removal. However the 20:1 recirculation ratio used could prove uneconomical on a full scale plant. Subsequent work using plastic filter media ('Flocor' being preferred) indicated that at pilot-scale levels a similar 80 percent BOD_5 removal could be achieved using two filters in series each loaded at 2.8 kg/m^3d and having recirculation ratios of 9:1. The savings in filter volume are therefore considerable. In both trials the effluent stream used comprised spent fermentation medium with a BOD_5 of approximately 10000 mg/ℓ and other yeast plant waste waters giving a combined BOD_5 of 7000-8000 mg/ℓ.

Treating yeast plant effluent diluted to 1800 mg/ℓ with water, using an aerobic trickling filter - 82.6 percent of the BOD_5 was removed along with 56.3 percent of the effluent carbohydrate (Sharkov, 1970). Both the volatile fatty acid and aldehyde concentrations of the waste water increased during treatment, the former indicating some anaerobic activity in the filter.

Rotating Disc Contactor

In a series of trials (Botuk *et al*, 1975), the effluent from a bakers' yeast and ethanol plant was treated using rotating disc contactors. This process is an aerobic contact one whereby a biomass is allowed to grow on discs arranged along a spindle. The row of discs is partially submerged in the effluent to be treated and as the spindle rotates the biomass is alternatively contacted with the waste organic material and atmospheric oxygen. In the trials the yeast and alcohol plant effluent was diluted to <1500 mg $BOD_{TOTAL}/ℓ$ and loaded on the discs at 0.05 kg BOD_{TOTAL}/m^2d. Treatment efficiency was 86 percent removal of BOD.

2.2.3 Anaerobic Biological Treatment of Yeast Plant Effluent

Anaerobic digestion technology has been applied to the

treatment of this waste water with generally good results. The digester configuration used in the studies were of the stirred and heated type.

Removal of Organic Material

COD removals of 77 percent for spent nutrient and 6 percent for mixed yeast plant wastes have been reported at retention times of 6 to 9 days (Stander *et al*, 1971). The above substate concentrations were 77000 mg COD/ℓ and 22000 mg COD/ℓ respectively. Twenty to 30 percent of the COD of yeast plant effluent has been shown to be resistant to biological degradation either anaerobic or aerobic (Stander *et al*, 1971; Hansford & Richtor, 1975). Regardless of the seeding and acclimation techniques used, the biologically resistant fractions remained even after a period of up to 18 months. Therefore the above removals represent at least 85 percent removal of the degradable COD fraction.

The effect of organic loading rate on removal of organic material is presented in Table 2.5. Increased substrate concentrations and decreased hydraulic retention times, both of which lead to increased loading rates gave rise to reduced fractional removals.

Doubling the loading rate from 0.8 to 1.6 kg BOD_5/m^3d reduced the fraction of BOD_5 removed from 0.90 to 0.79 (Trubnick & Rudolfs, 1948) and the removal of volatile solids fell from 0.86 to 0.72 when the loading rate was approximately trebled from 0.72 kg VS/m^3d (Ling, 1961). In apparent contradiction are the relatively constant removals of 95 percent (Sen & Bhaskaran,1962) and 75 percent (Botuk *et al*, 1973) over loading rate ranges of 0.74 to 3.0 kg BOD_5/m^3d and 1.5 to 2.5 kg BOD_5/m^3d respectively. In the former case, the BOD_5 removal did fall slightly to 91 percent when the organic loading rate was increased to 3.76 kg BOD_5/m^3d.

Maximum loading rates on digester biomass giving rise to BOD_5 removals of 70 to 80 percent have been reported as

4.8 and 8.8 kg VS/m^3d for digester volatile solids of concentrations of 10 and 20 percent respectively (Sonoda & Tanaka, 1963).

TABLE 2.5 : Removal of Organic Matter By Anaerobic Digestion

Loading rate kg BOD_5/m^3d	Removal %	Source
0.8	90 BOD_5	) Trubnick &
1.6	79 BOD_5	) Rudolfs (1948)
0.72*	86 VS	)
2.1 *	72 VS	) Ling (1961)
0.74 to 3.0	95 + 1 BOD_5	) Sen & Bhaskaran
3.76	91 BOD_5	) (1962)
1.5 to 2.5	75 BOD_5	Botuk *et al* (1973)

* VS not BOD_5

Gas Production

In most studies to date the digester gases, specifically the methane fraction have been considered merely a useful by-product of BOD_5 removal. Because of this emphasis on organic matter removal, less detailed data on gas production and composition is available in the literature.

Some data for gas production in the anaerobic digestion of yeast plant effluent is presented in Table 2.5a. There appears to be a decrease in gas production as the loading rate on the digester increases (Trubnick & Rudolfs, 1948) however one study (Sen & Bhaskaran, 1962) found that the decrease was not significant at loading rates below 3.01 kg BOD_5/m^3d but a large reduction occurred at 3.76 kg BOD_5/m^3d, the highest loading rate tested. These data follow a similar trend to the decrease in percentage organic matter removal. In addition to the decreasing total gas production at higher loading rates, methane

content of the gas also decreases (Sen & Bhaskaran, 1962; Hansford & Richtor, 1975) indicating a stressing of the anaerobic digestion process and the production of cellular intermediate compounds rather than methane.

Due to the lack of detail in the gas production data it is not possible to calculate the methane production in terms of the theoretical maximum as described by McCarty (1964). However, Stander *et al* (1971) quote their gas production as being 70 percent of the maximum, presumably on the same basis.

TABLE 2.5a: Gas Production from the Anaerobic Digestion of Yeast Plant Effluent

Loading Rate kg BOD_5/m^3d	Gas Production	CH_4 content %	Source
0.8	500ℓ/kg VS added	73.9)	Trubnick
1.6	430ℓ/kg VS added	73.9)	& Rudolfs (1948)
1.25 to 2.65	500ℓ/kg BOD_5 added	-	Merkel (1959)
3.76	137ℓ/kg BOD_5 added	49	Sen & Bhaskaran (1962)
0.26 kg COD/kg VSS.d	7500-8000ℓ/d	-	Stander *et al* (1971)
1.5 to 2.5		56 to 72	Botuk *et al* (1973)

Environmental Considerations

Two of the important parameters for aerobic digestion to proceed are a pH in the range 6.6 to 7.6 and an absence of toxic compounds (McCarty, 1964). Yeast plant effluent contains components which can adversely affect both of the above environmental requirements. It has a low pH of 4.6 - 5.5 initially, and a carbohydrate concentration of up to 1.5 percent (w/v), which is readily catabolised to organic acids by the fermentative bacteria and thereby

depressing the pH. Furthermore the presence of inorganic sulphates, due to the addition of nutrient supplements to the yeast propagation medium, may be reduced to more or less toxic sulphides in an anaerobic digestion environment.

There is some contradiction in the literature over pH maintenance in the digestion of yeast plant effluent. Adjustment of feed pH to 7.0 using sodium hydroxide was found necessary to maintain digester conditions in a favourable pH range (Ling, 1961) but no buffering was required when effluent recirculation was practised on a pilot scale digester (Stander *et al*, 1971). In a third case, buffering of pH was required at loading rates below 5 kg COD/m^3d whereas at higher rates there was sufficient buffer capacity to maintain pH at a favourable level.

An amount of sulphate in the digester feed material of 2000 mg/ℓ, which is in the normal range for yeast plant effluents, did not adversely affect the rate of digestion (Stander *et al*,1971); in fact,addition of sulphate to artificially increase the concentration to 18000 mg/ℓ in the feed, giving rise to sulphide levels of 400 mg/ℓ in the digester were not deleterious. The limit sulphide concentration giving rise to severe inhibition of 150 mg/ℓ (McCarty, 1964) was exceeded possibly due to the build up of some sulphide tolerance by the digester biomass over the period of the studies.

2.2.4 Mixed Anaerobic and Aerobic Biological Treatment of Yeast Plant Effluent

Figure 2.2 outlines a five stage waste treatment system from a bakers' yeast plant in Poland. The stages are

(a) methane fermentation in a two-stage anaerobic digestion phase based on initial studies reported by Tomczynska (1971);

(b) solids removal by sedimentation;

(c) aerobic trickling filter treatment of the sedimentation supernatant;

(d) further sedimentation of the filter effluent, and,

(e) aerobic lagoon treatment before ultimate discharge.

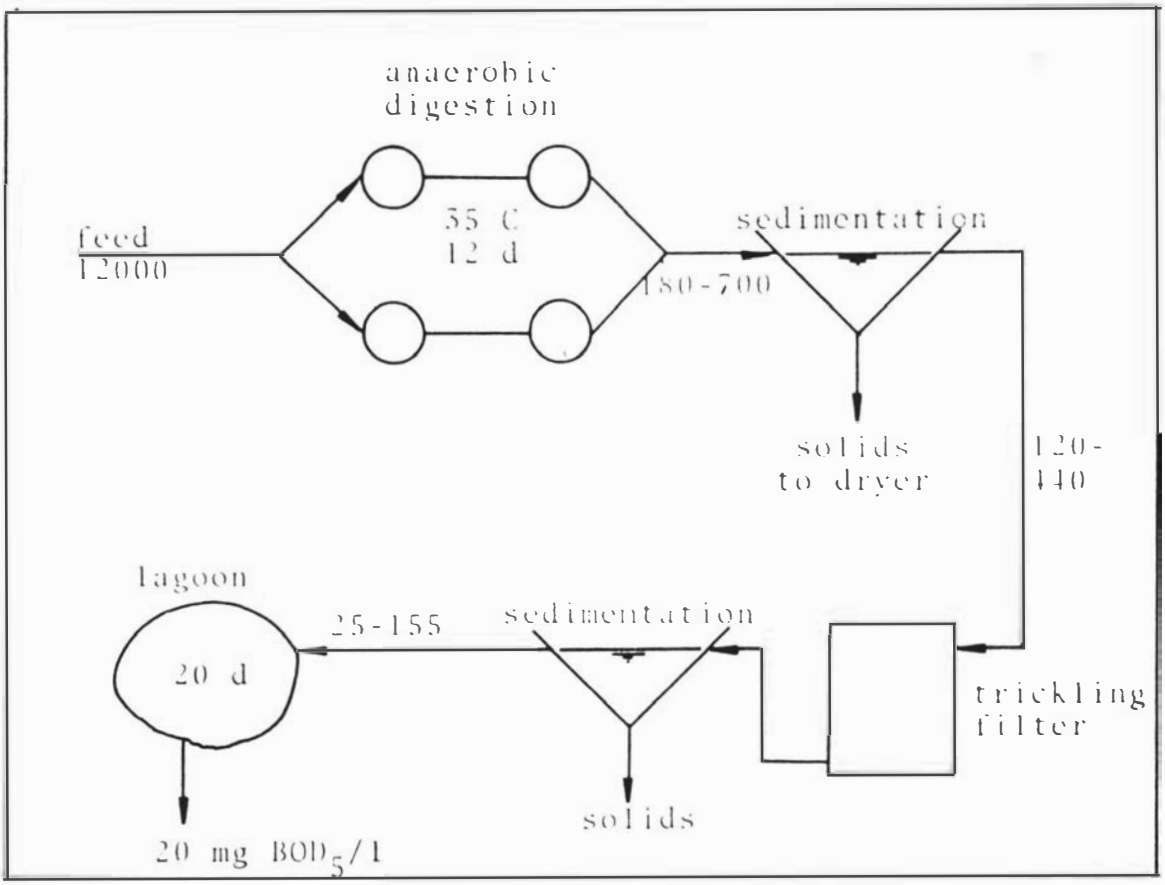


FIGURE 2.2 : Yeast Plant Waste Water Treatment System
(Based on Tomczynska, 1971).
All values in mg BOD_5/ℓ except where noted

Summary

The major polluting waste stream arising from the production of bakers' yeast is the spent fermentation medium from the final propagation stage. Other waste streams are the sludge from molasses clarification and the cooling and plant wash waters. Both these latter waste streams could be relatively simply separated from the spent nutrient for separate disposal. The molasses sludge could be disposed of by transportation to a landfill site and the cooling and wash waters sent to a municipal sewerage system.

For a commercial yeast plant located within a municipality, separate disposal of the molasses sludge rather than diluting it and washing it down the sewer would reduce by fifteen percent the disposal costs for BOD_5 on the plant. On-site treatment of the spent fermentation medium as a separate stream would further reduce this cost.

The fermentation medium has been successfully treated for BOD_5 removal by both aerobic and anaerobic processes. While the aerobic treatments successfully reduced the BOD_5 by up to 99 percent these systems were operating at the upper limit of substrate concentration generally associated with aerobic biological treatment. Further, the most effective aerobic treatment process, activated sludge, is a highly sophisticated process requiring close control of operating parameters and high operating costs of oxygen transfer equipment. There is also the problem of disposal of the biomass into which much of the organic material in the waste stream is converted. Since overall yeast production economics are dependent on waste disposal costs, such high costs would disadvantage aerobic treatment systems compared to the anaerobic ones.

Yeast plant effluent has also been shown to be amenable to anaerobic digestion with high organic material removal efficiencies. Anaerobic processes in general are more suited to the high BOD_5 concentrations found in yeast effluent. In a properly operated anaerobic digester organic material is converted to gaseous products, principally methane with consequently lower sludge disposal costs. Due to the higher influent substrate concentrations able to be applied to an anaerobic digestion system, a smaller treatment system is possible.

2.3 The Anaerobic Waste Treatment Process

In anaerobic waste treatment, complex organic materials in the waste stream are converted to methane and carbon dioxide in the absence of molecular oxygen. The process, which occupies an essential niche in natural self purification processes, was first used in the waste treatment field for the stabilisation of sewage sludges, but has more recently been applied to the treatment of higher strength industrial wastes (Tebbutt, 1971). McCarty (1964) has stated five important advantages over aerobic digestion. These are :

(1) a higher degree of waste stabilisation;

(2) a lower yield of biological solids;

(3) lower nutrient requirement due to the reduced solids production;

(4) no requirement of oxygen;

(5) production of methane gas which is a useful product.

During digestion, organic material is converted into methane and carbon dioxide and, as up to 90 percent of the energy in the waste is retained by the gas, little is available for growth and cell yields are lower than the 50 percent possible in aerobic systems (McCarty, 1964). Anaerobic digestion in the rumen of some animals under normal conditions stops at acid production which is then utilised by the animal for growth and energy.

2.3.1 Theoretical Aspects of Methane Production in Anaerobic Digestion

Despite its widespread and growing use there has been, until recent years, a poor understanding of the process

fundamentals. Detailed reviews of the process microbiology, biochemistry, chemistry and environmental factors have been published (McCarty, 1964; Kirsch & Sykes, 1971; Hobson *et al*, 1974; Torien & Hattingh, 1969; Kotzé *et al*, 1969; Mah *et al*, 1977)

One major problem appears to be the great difficulty in isolating pure cultures of the methanogenic and acetogenic organisms.

Environmental factors required for sustained anaerobic digestion are

(1) pH in the range 5 to 8, however activity declines sharply below pH 6.6;

(2) an absence of oxygen;

(3) sufficient nutrients, and,

(4) an absence of toxic materials.

In addition there appear to be two groups of anaerobic digestion organisms which operate at different temperature ranges; thermophilic organisms at temperatures of 45 to 70^{o}C and mesophilic ones at 30 to 40^{o}C (Mah *et al*, 1977; Bryant, 1979).

In many natural environments such as swamps and organic sediments in fresh water and marine ecosystems, the anaerobic decomposition of organic matter plays an important role in the cycle of nutrients. Anaerobic digestion is also found to some extent in the gut of ruminants, and much of the literature on the process is related to reactions in rumen fluid.

The microbiology of anaerobic digestion was generally thought to be divided into two groups (Torien & Hattingh, 1969). One group fermented complex or polymeric compounds to fatty acids, alcohols, CO_2, NH_3 and sulphide but was not capable of producing the end product methane. The second group were the methane formers which obtain energy for growth

by fermenting the products of the first group to methane and carbon dioxide. Recent research however has shown this two stage scheme to be unsatisfactory (Bryant,1979). Methanogenic bacteria are unable to catabolise organic acids other than acetic and formic, alcohols other than methanol; and the production and utilisation of hydrogen plays a control role in the fermentation (Bryant, 1979; Mah *et al*, 1977).

A modified scheme for the conversion of carbohydrates to methane and CO_2 is presented in Figure 2.3.

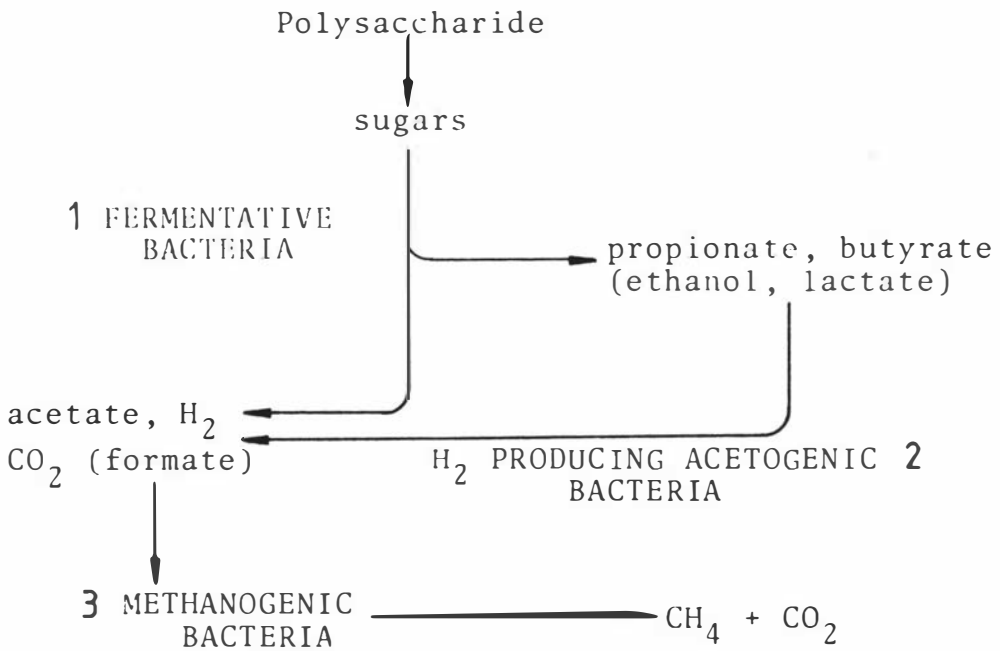


FIGURE 2.3 : Generalised scheme of the three stages involved in anaerobic fermentation (Bryant, 1979).

In the above scheme in addition to the previously mentioned fermentative and methanogenic groups of bacteria, there is an intermedidate group known as hydrogen producing

acetogenic bacteria. This last group obtain their energy for growth by producing acetate, H_2 and sometimes CO_2 from the products of initial fermentation (Bryant, 1979). While the three phases of the anaerobic processes can be considered separate (as in the scheme), the metabolism of each is dependent on the others (Bryant, 1979).

In the first stage of anaerobic degradation of a carbohydrate, the pathways illustrated in Figure 2.4 have been proposed (Hobson *et al*, 1974; Mah *et al*, 1977; McCarty, 1964; Torien & Hattingh, 1969; Bryant, 1979).

The polysaccharide is hydrolysed to sugars which are then fermented to pyruvate. Electrons designated as 2H are produced in the form of the reduced nicotinamide adenine dinucleotide (NADH). Pyruvate is then catabolised further to acetate, CO_2 and H_2 or the propionate, butyrate or ethanol (Bryant, 1979).

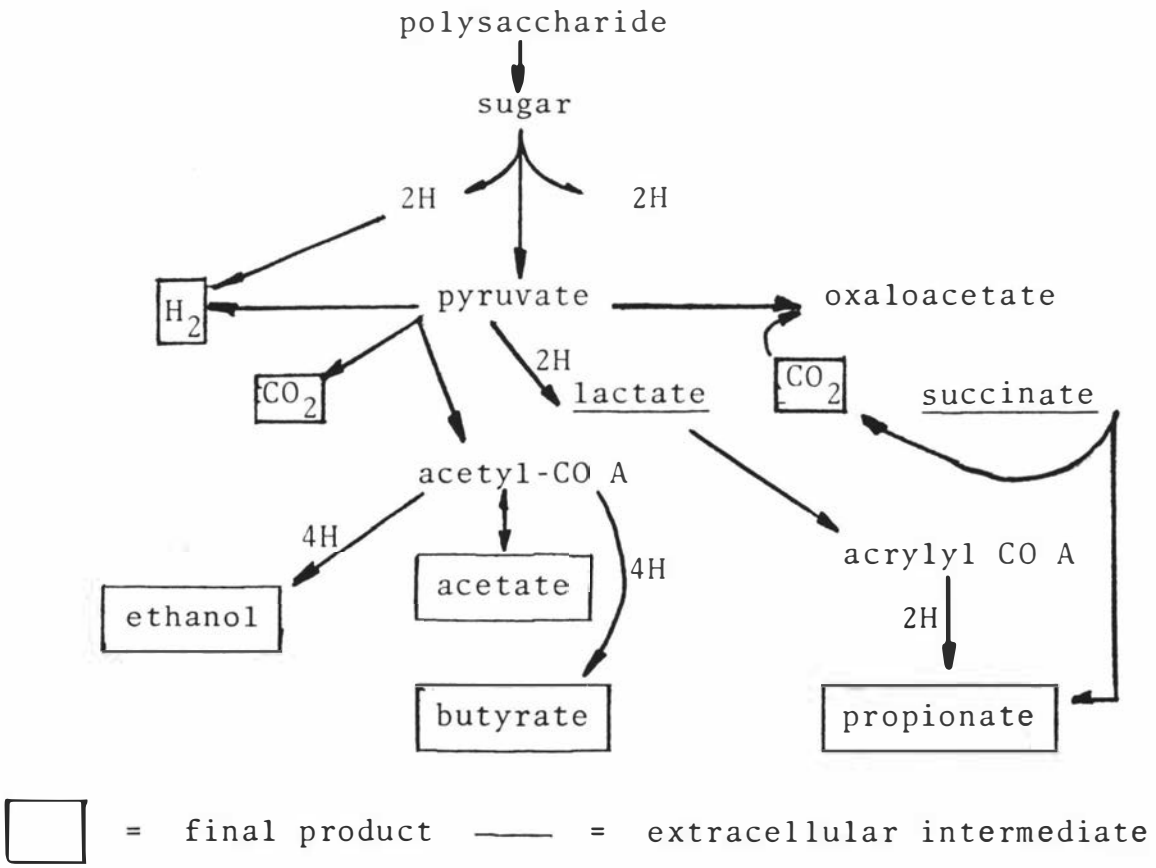


FIGURE 2.4 : Pathways involved in first stage fermentation of carbohydrate (After Bryant, 1979)

Recent studies of the metabolic interactions between the fermentative and H_2 - utilising methanogenic bacteria (Bryant, 1979) suggest that the concentration of H_2 in the ecosystem plays a central role in controlling the various product proportions by the fermentative bacteria, through the formation of the reduced NAD as in equation 2.3.

$$NADH + H^+ \rightleftharpoons H_2 + NAD^+$$

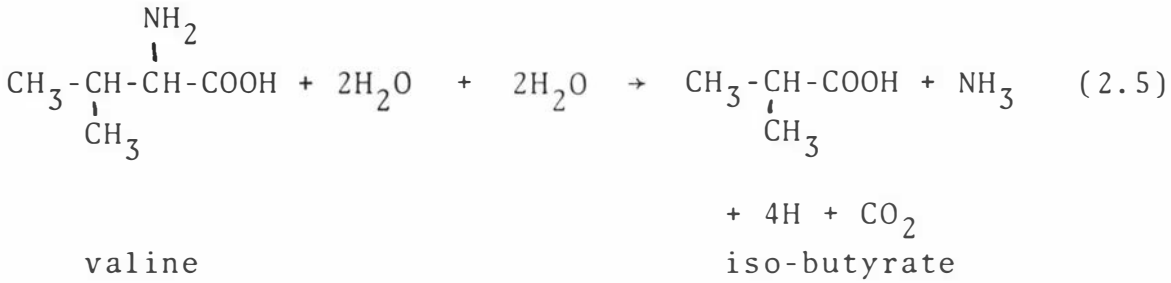
$$\Delta G'_o = + 17.97 \text{ kJ/mol.} \qquad (2.3)$$

$$K = 6.84 \times 10^{-4}$$

For degradation to proceed production of H_2 from NADH oxidation is essential. The equilibrium is however in the direction of NADH production unless the H_2 partial pressure is extremely low, as it is in the presence of H_2 utilising methanogenic bacteria. When the system is stressed as in the case of overloading with organic matter or reduced retention time there is a tendency for the electrons of NADH generated in fermentation to be utilised in catabolism of pyruvate to the reduced products especially propionate rather than acetate, CO_2 and H_2.

The fermentative bacteria also use many other compounds. Lipids are hydrolysed, unsaturated fatty acids hydrogenated and long chain fatty acids ß-oxidised to produce volatile acids for subsequent conversion in the latter stages (Andrews & Pearson, 1965). The branched volatile acids iso-valerate and iso-butyrate may also be produced directly via Strickland reactions (equations 2.4 and 2.5) from some amino acids.

$$\underset{\text{leucine}}{CH_3\text{-}\underset{CH_3}{\underset{|}{CH}}\text{-}CH_2\text{-}\underset{H}{\underset{|}{\overset{NH_2}{\overset{|}{C}}}}\text{-}COOH} + 2H_2O \rightarrow \underset{\text{iso-valerate}}{CH_3\text{-}\underset{CH_3}{\underset{|}{CH}}\text{-}CH_2\text{-}COOH} + NH_3 + 4H + CO_2 \qquad (2.4)$$



$$CH_3\text{-}\underset{\displaystyle CH_3}{\overset{\displaystyle NH_2}{CH}}\text{-}CH\text{-}COOH + 2H_2O + 2H_2O \rightarrow CH_3\text{-}\underset{\displaystyle CH_3}{CH}\text{-}COOH + NH_3 + 4H + CO_2 \qquad (2.5)$$

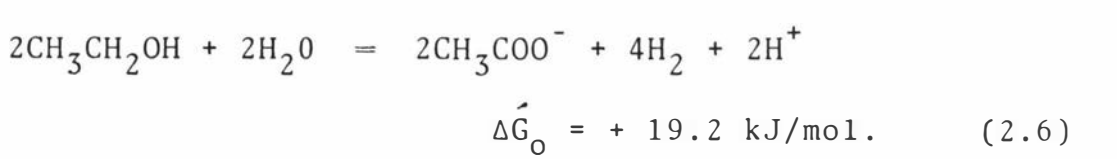
valine iso-butyrate

The products of first stage degradation apart from CO_2, H_2 and acetate are mainly propionate and longer chain volatile acids and are anaerobically oxidised to acetate or acetate and CO_2 (McCarty, 1964). Little is known about the electron sink for reactions and in the known cases it is H_2. More than one species of acetogenic bacteria may be necessary to convert some substrates such as odd numbered fatty acids. For instance one species may beta-oxidise an acid to acetate, propionate and H_2 and another species may be necessary for oxidative decarboxylation of propionate to acetate, CO_2 and H_2 (Bryant, 1979).

The maintenance of an extremely low H_2 partial pressure is necessary for these acetogenic bacteria to grow and catabolise substrates.

In pure cultures the free energy change does not favour catabolism of the fatty acids, but favourable energetics result when cocultured with the methanogen.

Direct evidence for the obligate H_2-producing acetogenic phase was provided by Bryant *et al* (1967) when the 'species' *Methanobacillus omelienskii* was resolved into two species syntrophically converting ethanol to methane. The oxidation of ethanol to acetate is thermodynamically unfavourable (equation 2.6) unless coupled with the H_2 utilising bacteria.



$$2CH_3CH_2OH + 2H_2O = 2CH_3COO^- + 4H_2 + 2H^+$$

$$\Delta G'_o = +19.2 \text{ kJ/mol.} \qquad (2.6)$$

Other evidence is provided by the isolation of a fatty acid catabolising bacteria, apparently a new species, that degrades even carbon numbered fatty acids butyrate, caproate, caprylate to acetate and H_2. Odd numbered acids valerate and heptanoate were degraded to acetate, propionate and H_2. The bacteria could only be grown in syntrophic association with a hydrogen utilising desulphovibrio or methanogen. No other electron donors, acceptors or energy sources were utilised (McInery *et al*, 1979). This species degrades butyrate according to equation 2.7.

$$CH_3CH_2CH_2COO^- + 2H_2O \rightleftharpoons 2CH_3COO^- + H^+ + 2H_2 \qquad (2.7)$$
$$\Delta G'_o = + 48.1 \text{ kJ/mol.}$$

A new genus and species of bacteria *Syntrophobacter wolini* which degrades propionate has been isolated in coculture with the hydrogen utilising *Desulphovibrio* species which cannot utilise organic acids other than formate. The free energy change for the propionate degradation is + 76.1 kJ/mol (Boone & Bryant, 1980).

Three strains of a new genus and species of bacteria, *Syntrophomonas wolfei* were isolated and characterised by McInery *et al* (1981). This organism beta-oxidises C_4 to C_8 saturated fatty acids to acetate, or acetate and propionate using protons as the electron sink. The organism could only be isolated in coculture with H_2 utilising *Desulphovibrio* or methanogen and had generation times of 54 to 84 hours. Lactate may also be completely degraded to carbon dioxide and methane by a syntrophic association of *Methanosarcina barkerii* and *Desulphovibrio* species (McInery and Bryant, 1981).

Methanogenic bacteria are only directly involved in the third stage of anaerobic digestion, although their

importance to the H_2 producing acetogenics has already been discussed. They are the only organisms capable of utilising electrons and breaking down acetate in the absence of light, or other electron acceptors such as sulphate or nitrate. In the absence of the methanogens, organic acids from the fermentative stage of anaerobic degradation would accumulate yielding an energy content almost as great as the initial organic waste (Bryant 1979).

The methanogenic bacteria appear to be a unique group of organisms with common energy producing metabolisms, the stoichiometry of which is presented below.

Energy Sources of Methanogenic Bacteria
(After Bryant, 1979)

1. All species

$$4H_2 + HCO_3^- + H^+ \rightleftharpoons CH_4 + 3H_2O$$

$\Delta G'_o$ = -32.4 kcal/mol.

2. Many species

$$4HCOO^- + H_2O + H^+ \rightleftharpoons CH_4 + 3HCO_3^-$$

$\Delta G'_o$ = -31.2 kcal/mol.

3. Few species

$$CH_3COO^- + H_2O \rightleftharpoons CH_4 + HCO_3^-$$

$\Delta G'_o$ = -7.4 kcal/mol.

4. One species

$$4CH_3OH \rightleftharpoons 3CH_4 + HCO_3^- + H^+ + H_2O$$

$\Delta G'_o$ = -75.2 kcal/mol.

All organisms studied were able to use H_2 to reduce carbon dioxide to methane. Although some 70 percent of the methane produced from the organic material was derived from the methyl group of acetate (McCarty, 1964) only a few species

are known to reduce acetate in this way. These include *Methanosarcina barkerii*, the only example to be isolated in pure culture (McInery & Bryant, 1981). This latter is also the only organism known to convert ethanol to methane directly.

Work is being carried out to further elucidate the metabolism of the methanogenic bacteria. The contribution of each of the organic acid intermediates has recently been reported (Mackie & Bryant, 1981). Although the acids of C_3 and greater are converted to methane via acetate it was found from radioactive tracer studies that propionate and butyrate respectively contributed 13 to 17 and 7 to 9 percent of the methane.

The yield of methane has been predicted stoichiometrically from a knowledge of the chemical compositions of the substrate. (equation 2.8).

$$C_nH_aO_b + (n-\frac{a}{4} - \frac{b}{2})H_2O \rightleftharpoons (\frac{n}{2} - \frac{a}{8} - \frac{b}{4})CO_2 + (\frac{n}{2} + \frac{a}{8} - \frac{b}{4})CH_4 \quad (2.8)$$

(Buswell & Mueller, 1952)

In waste treatment situations where the substrate is not a pure compound, the theoretical maximum methane yield is 0.35 m^3 per kilogram of COD of ultimate BOD stabilised. (McCarty, 1964).

In summary it is noted that anaerobic digestion involves a complex interaction between three groups of organisms namely;

(1) the fermentative organisms which degrade complex molecules to simpler compounds mainly pyruvate, organic acids, hydrogen and carbon dioxide;

(2) an acetogenic hydrogen producing group which degrade higher organic acids, lactate, ethanol and perhaps other compounds to acetate and hydrogen,

(3) a methanogenic group all of which reduce carbon dioxide to methane, a few reduce acetate and one

species reduces ethanol to methane.

The metabolic activities of the latter two groups is largely unknown and isolation of pure cultures of the organisms is difficult. Hydrogen concentration plays an essential role in overall conversion of organic material to methane. Its concentration must be kept at an extremely low level to allow the acetogenic organisms to have favourable thermodynamics for their catabolism of substrates.

2.3.2 Anaerobic Process Designs

There are several designs of anaerobic processes based on the kind of reactor used. These are;

(1) the anaerobic digester which is a stirred tank reactor;

(2) contact digesters also known as anaerobic activated sludge reactors which comprise a reaction vessel and a cell separation phase for biomass recycle, and,

(3) the anaerobic filter which is similar to the aerobic trickling filter.

More recently a fourth configuration has been tried, that of a fluidised bed of inert material attached to which is the anaerobic digestion biomass. Figure 2.5 illustrates schematically the types of anaerobic digestion reactors.

Conventional systems usually comprised circular concrete tanks with fixed or floating covers. Feed was on a continuous or intermittant basis and retention times of 30 to 60 days were common. These first anaerobic digesters were unmixed and unheated and consequently were very inefficient. Mixing, either mechanically or by gas or contents recirculation, and heating by external or internal heat exchangers lead to reduced retention times of 10 to 20 days and the present generation of so-called high-rate digesters. Although anaerobic digesters find most applications in the treatment of primary sewage sludges which are stabilised in approximately 15 d. at 30 to 35^{o}C, some other wastes are amenable to such treatment, such as secondary sewage sludges, and some industrial wastes (Grady & Lim, 1980).

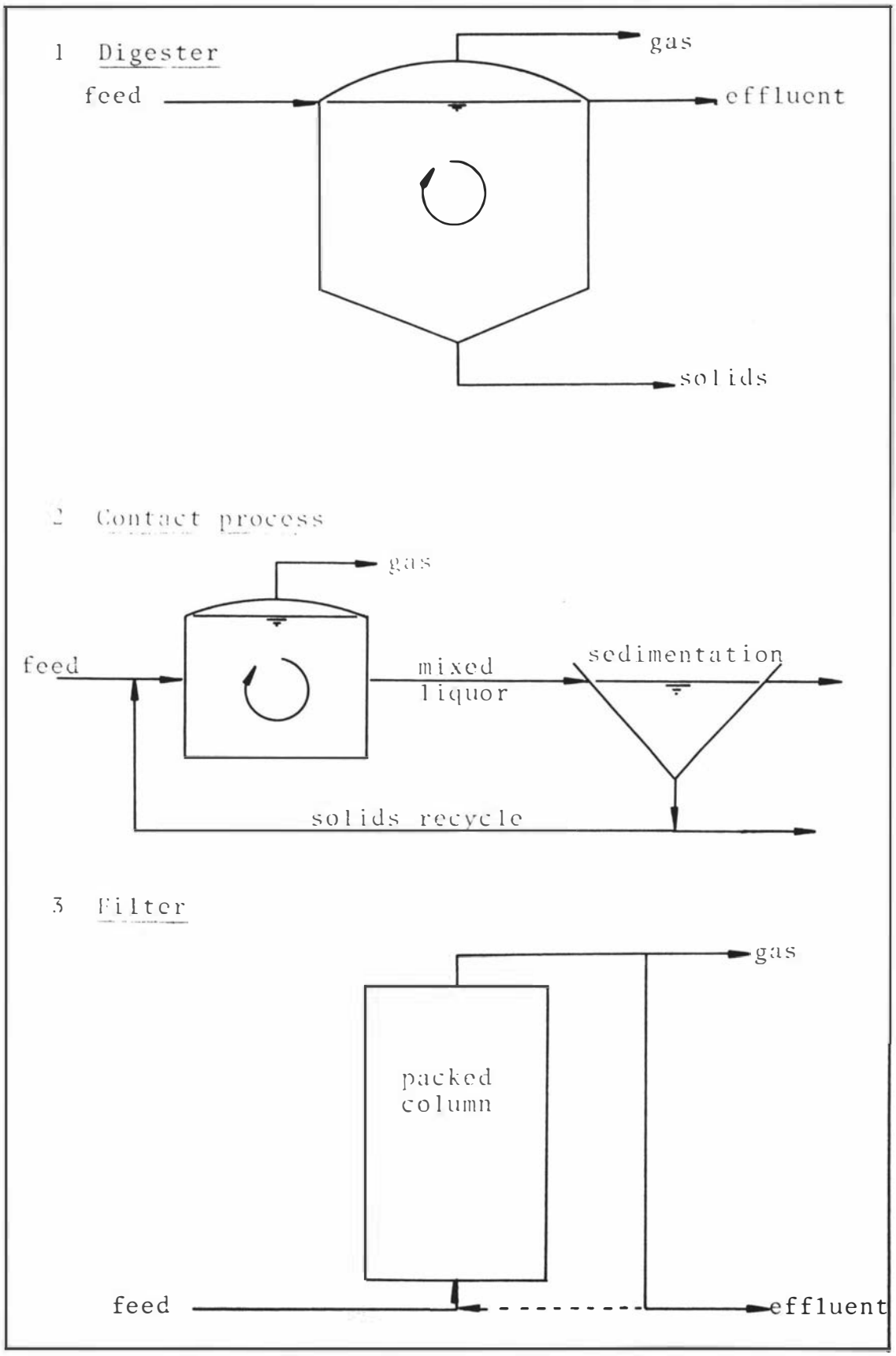


FIGURE 2.5: Schematic diagram of some anaerobic waste treatment configurations.

The gases produced by anaerobic digesters are generally burnt to produce heating for the digester and feedmaterial, power for a treatment facility, or vented.

The anaerobic contact process was developed in response to a need to maintain a high concentration of active biomass to treat relatively dilute waste streams. This configuration is analogous to the activated sludge process and was first applied to the treatment of meat packing wastes (Schroepfer *et al*, 1955). Such a waste stream is relatively dilute compared to sewage sludges and hydraulic retention times of the order of 12 hours are possible using this process. The main disadvantage of this system is associated with solids separation for recycle, requiring degasification of the sludge and/or the use of chemical flocculants especially in the treatment of soluble wastes and the requirement for external heating since the dilute wastes generate insufficient methane to maintain the 35^{o}C operating temperature. Although this process has been applied to some industrial wastes its acceptance has been limited even though it is more economical than activated sludge treatment (Pffefer, 1979).

In order to overcome the problems associated with solids recovery of the anaerobic contact process, two digester configurations utilising a fixed biomass have been developed. These are the anaerobic expanded bed reactor and the anaerobic filter. Both systems employ an inert support medium to which the biomass adheres thereby eliminating the need for solids recycle and allowing the waste water to pass through the system at short retention times.

The expanded bed reactor utilises a biomass attached to a granular support which is fluidised in operation thereby providing a large surface area and rapid treatment of the waste (Jeris *et al*, 1977). The system has been used to treat dilute waste waters of less than 600 mg COD/ℓ (Switzenbaum, 1978) at retention times of several hours and near ambient temperature. This work led to the possibility of using the process for the treatment of domestic sewage without

the need for the costly aeration of conventional aerobic systems. It has also been applied to the treatment of medium strength industrial wastes of up to 5000 mg COD/ℓ at retention times of 2.6 to 9.3 h (Boening & Larsen, 1981) Although having low operating costs, the expanded bed process is extremely sensitive to variations in flow rate hence requiring a continuous feed and constant monitoring.

The anaerobic filter on the other hand is the simplest of the anaerobic digesters, comprising simply a packed bed of support medium in a cylindrical vessel and pumping to pass the waste water up through it. Its ease of operation and very low operating costs (Pfeffer, 1979) and relative immunity to shock loading (Jennett & Dennis, 1975; Jennett de Walle, 1976) and interruptions in feed rate (Taylor & Burm, 1975), indicate its potential efficacy in the pre-treatment of a high strength industrial waste, low in suspended solids such as yeast plant effluent.

2.4 The Anaerobic Filter

The use of an inert support medium to maintain a high concentration of biomass in an anaerobic digestor without washout at high hydraulic loadings, was initiated several decades ago. The addition of asbestos fibres to a digester treating dairy plant effluents was reported by Buswell, 1936. The use of a packed bed configuration occurred some 25 years later with the description of an upflow percolating filter (Dewes, 1960). This latter system was used to anaerobically treat several industrial effluents including those arising from the processing of dairy products, and the production of wood pulp and citric acid. Acid fermentation in the filter had a 'rapid purifying effect' on the waste waters and residual impurities could be removed by chemical treatment. The ground-breaking work on an anaerobic digester configuration which had minimal maintenance requirements and sludge handling problems, and had the potential to operate at near ambient temperatures, was carried out by Young and McCarty (1967).

Following the initial study, the anaerobic filter has been applied to the treatment of a variety of waste streams both synthetic (Plummer *et al*, 1968; Mueller & Mancini, 1975; Kahn & Siddigi, 1976), and real. These latter included the waste waters arising from industrial activities of : pharmaceutical companies (Jennett & Dennis, 1975; Seeler & Jennett, 1978; Sachs *et al*, 1978); a tannery (Arora *et al*, 1975); a brewery (Foree & Lovan, 1971, 1972); the petrochemical industry (Hovious *et al*, 1971) shellfish processing (Hudson *et al*, 1978) and milk processing (Peterson, 1975). In association with domestic wastes treatment, the anaerobic filter has been applied to the treatment of waste activated sludge (Haug *et al*, 1977) from sewage treatment, the leachate from sanitary landfill sites (de Walle & Chian, 1977; Johanson & Carlson, 1976), and raw sewage (Genung *et al*, 1979).

With removal of organic material as a secondary concern, the anaerobic filter has been utilised in the denitrification of waste waters (St Amant & McCarty, 1969; Tamblyn & Sword 1969; Anderson & Ibrahim, 1978) and to recovery of ammonia (Trulsson, 1979).

2.4.1 Filter Design

The design of the anaerobic filters used in most studies has followed the pattern of those used in the first study by Young & McCarty, (1967). Experimental units are cylinders of a high aspect ratio, mounted vertically and fed from the bottom. Packing material has varied in both composition and packing density. Rock fillings give rise to void ratios of the order of 0.45 (Young & McCarty, 1967; Jennett & Dennis, 1975; Sachs *et al*, 1978; Hobson *et al*, 1978). Void ratios of 0.95 and greater have been achieved with the use of plastic support media of similar type to aerobic trickling filter medium. In addition some novel packing materials have been used such as oyster shells (Hudson *et al*, 1978).

Modifications to this basic design have been made in several studies. Multiple filter units in series have been

successfully used to extend filter capacity (El-Shafie & Bloodgood, 1973). An attempt made to separate the acid formation and methanogenic stages by using an acidic stirred tank reactor followed by an anaerobic filter met with only limited success (Norman & Frostell, 1977). Replacement of an entrapment medium by long narrow tubes, the insides of which comprise the support surface, has also been reported (van den Berg & Lentz, 1980).

The scale of most anaerobic filter studies has been limited to laboratory or bench size units. However, at least two commercial scale units have been commissioned (Taylor & Burm, 1975; Witt *et al*, 1979).

2.4.2 Operational Conditions

Steady state operational data from the literature is presented in Table 2.6. The principal input parameters are the influent substrate concentration and the hydraulic retention time, the latter based on initial filter void volume. Together these parameters are used to define the organic loading on the system in terms of kg organic material/ m^3.d.

In Figure 2.6 the range of influent substrate concentrations and hydraulic retention times tested are presented. The lines are points of equal organic loading rate.

Although the maximum substrate concentrations and hydraulic retention times used are 16000 mg COD/ℓ and 10.5 d respectively (Figure 2.6), the bulk of the trials have been carried out under conditions of less than 6000 mg COD/ℓ and 5d hydraulic retention time.

TABLE 2.6: Steady State Performance Data for some Anaerobic Filters

Influent		HRT	Loading rate		Temp.	Effluent								
						COD		BOD_5		Alkalinity	VFA	pH	methane in gas	
COD	BOD_5		COD	BOD			Removal		Removal	mg/ℓ	mg/ℓ			
mg/ℓ		d	kg/m^3d		°C	mg/ℓ	%	mg/ℓ	%	(1)	(2)		%	
1500	*	0.63	2.38	*	25	100	92.1	25	98.4	*	15	*	72.0	Young & McCarty (1967)
1500	*	0.32	4.76	*	25	110	91.5	35	97.5	*	22	*	72.0	
1500	*	0.16	9.38	*	25	300	79.3	225	84.3	*	149	*	72.0	
1500	*	0.08	18.75	*	25	600	36.7	525	63.2	*	225	*	72.0	
3000	*	1.26	2.38	*	25	170	93.4	20	99.2	*	18	*	72.0	
3000	*	0.63	4.76	*	25	280	88.4	130	95.5	*	78	*	72.0	
3000	*	0.16	18.75	*	25	845	63.0	705	75.4	*	185	*	72.0	
1500	*	0.63	2.38	*	25	20	99.4	20	98.7	*	*	*	72.0	
1500	*	0.32	4.76	*	25	135	90.5	135	90.8	*	*	*	72.0	
1500	*	0.16	9.38	*	25	310	79.0	310	79.4	*	*	*	72.0	
1500	*	0.08	18.75	*	25	470	68.4	470	68.5	*	*	*	72.0	
3000	*	1.26	2.38	*	25	36	98.6	36	98.6	*	*	*	72.0	
3000	*	0.63	4.76	*	25	230	92.0	230	95.0	*	*	*	72.0	
6000	*	0.63	9.38	*	25	124	97.7	124	97.8	*	*	*	72.0	
6000	*	0.32	18.75	*	25	772	86.9	772	84.0	*	*	*	72.0	
546	975	3.46	1.61	0.61	35	*	86.0	*	*	3760	*	*	56.0	Plummer *et al* (1968)
2405	1548	2.50	3.79	0.41	35	*	68.0	*	*	2790	1545	*	52.0	
4850	3705	0.88	7.01	0.53	35	*	45.0	*	*	2750	2460	*	37.0	
5000	3890	0.54	10.20	0.38	35	*	35.0	*	*	2845	3355	*	32.0	
1000	*	2.00	0.22	*	25	45	95.5	*	*	270	36	6.5	*	Jennett & Dennis (1975)
1250	*	1.50	0.37	*	25	74	93.7	*	*	538	60	6.8	*	
1250	*	1.00	0.56	*	25	56	95.3	*	*	672	32	7.2	*	
4000	*	1.50	1.17	*	25	88	97.8	*	*	896	72	7.4	*	
4000	*	1.00	1.76	*	25	99	97.5	*	*	463	68	6.4	*	
4000	*	0.75	2.34	*	25	197	95.1	*	*	372	48	6.7	*	
4000	*	0.50	3.52	*	25	254	93.7	*	*	332	132	6.7	*	
8000	*	1.00	3.52	*	*	381	95.3	*	*	416	102	6.7	*	
16000	*	2.00	3.52	*	25	390	97.6	*	*	446	156	6.7	*	
10000	*	0.75	13.30	*	30	3300	70.3	*	*	*	*	7.0	62.0	El Shafie & Bloodgood (1973)
6600	7000	1.75	3.79	4.00	32	*	64.0	*	57.0	*	*	*	*	Taylor & Burm (1975)
2500	*	8.30	0.30	*	25	70	97.0	*	*	590	*	6.9	*	Peterson (1975)
2500	*	3.80	0.65	*	25	93	96.0	*	*	630	30	6.8	72.0	
2500	*	2.00	1.17	*	25	120	94.5	*	*	760	30	6.9	72.0	
2500	*	1.33	1.75	*	25	177	93.0	*	*	880	30	6.9	70.5	
1250	*	0.67	1.75	*	25	175	86.0	*	*	800	*	6.8	70.5	
625	*	0.33	1.75	*	25	170	72.0	*	*	730	30	6.8	70.5	
5000	*	1.33	3.50	*	25	355	93.0	*	*	930	30	6.9	70.5	
2500	*	0.67	3.50	*	25	350	86.0	*	*	890	*	6.9	70.5	
1250	*	0.33	3.50	*	25	350	72.0	*	*	820	30	6.9	70.5	
7500	*	1.33	5.25	*	25	665	91.0	*	*	1130	*	6.9	69.0	
3400	*	4.30	0.80	*	35	*	90.0	*	*	3000	*	*	65.0	Foree & Lovan (1972)
330	*	1.26	0.26	*	*	115	65.0	*	*	*	26	8.0	*	Arora *et al* (1975)
990	*	1.26	0.79	*	*	180	82.0	*	*	*	39	7.9	*	
2640	*	1.26	2.10	*	*	327	88.0	*	*	*	37	8.0	*	
4290	*	1.26	3.40	*	*	2314	46.0	*	*	*	47	7.9	*	
5610	*	1.26	4.45	*	*	4130	26.0	*	*	*	60	8.2	*	
9500	3000	2.00	4.75	0.63	32	2300	76.0	450	85.0	3500	300	7.1	70.0	Haug *et al* (1972)

Table 2.6:cont.

2500	*	0.70	3.57	*	32	*	64.0	*	*	*	*	*	*	
2500	*	1.90	1.32	*	32	*	76.0	*	*	*	*	*	*	Hovius *et al* (1971)
2800	*	1.50	1.87	*	37	*	90.0	*	*	*	*	*	*	
9500	4250	5.90	1.60	0.72	30	1330	86.0	155	96.3	*	*	7.40	85.2	
9500	4250	5.00	1.90	0.85	25	1500	84.2	235	94.5	*	*	7.80	87.6	van Velsen *et al* (1979)
9500	4250	2.40	4.00	1.77	30	950	90.0	90	97.9	*	*	7.60	86.3	
9500	4250	1.40	6.70	3.04	30	1755	81.5	475	88.8	*	*	7.90	86.9	
5260	*	9.40	0.56	*	33	270	95.0	*	*	750	35	6.95	80.0	
7140	*	10.50	0.68	*	33	170	96.0	*	*	775	25	7.20	70.0	
5400	*	6.60	0.82	*	33	250	95.5	*	*	550	15	7.00	*	Norman & Frostell (1977)
5500	*	4.70	1.17	*	33	240	95.5	*	*	430	15	7.00	65.0	
6800	*	3.10	2.19	*	33	350	95.0	*	*	330	15	6.80	*	
2000	*	1.50	0.56	*	35	500	75.0	*	*	*	*	7.20	*	Sachs *et al* (1978)
6000	*	1.50	1.67	*	35	4920	18.0	*	*	*	*	7.20	*	
2660	*	1.00	2.26	*	*	90	96.5	*	*	*	*	*	*	
1730	*	0.94	1.83	*	*	10	99.4	*	*	*	*	*	*	
2610	*	1.02	2.56	*	*	560	78.5	*	*	*	*	*	*	
1360	*	0.92	1.47	*	*	135	99.0	*	*	*	*	*	*	Anderson & Ibrahim (1978)
2110	*	0.33	6.40	*	*	42	98.0	*	*	*	*	*	*	
2500	*	0.33	7.50	*	*	400	84.2	*	*	*	*	*	*	
1760	*	0.34	4.92	*	*	170	99.0	*	*	*	*	*	*	
404	*	3.10	0.15	*	*	79	*	*	*	460	*	7.30	85.0	
357	310	1.60	0.25	1.24	*	70	*	58	*	460	*	7.20	88.0	Hudson *et al* (1978)
89	*	0.33	0.36	*	*	43	*	*	*	265	*	7.20	*	
404	*	2.51	0.18	*	*	284	*	*	*	460	*	7.20	62.0	
357	310	1.68	0.24	1.29	*	158	*	88	*	370	7	*	73.0	
89	*	0.35	0.34	*	*	43	*	*	*	240	*	7.13	*	

* no data available

(1) as $CaCO_3$

(2) total volatile acids as acetic

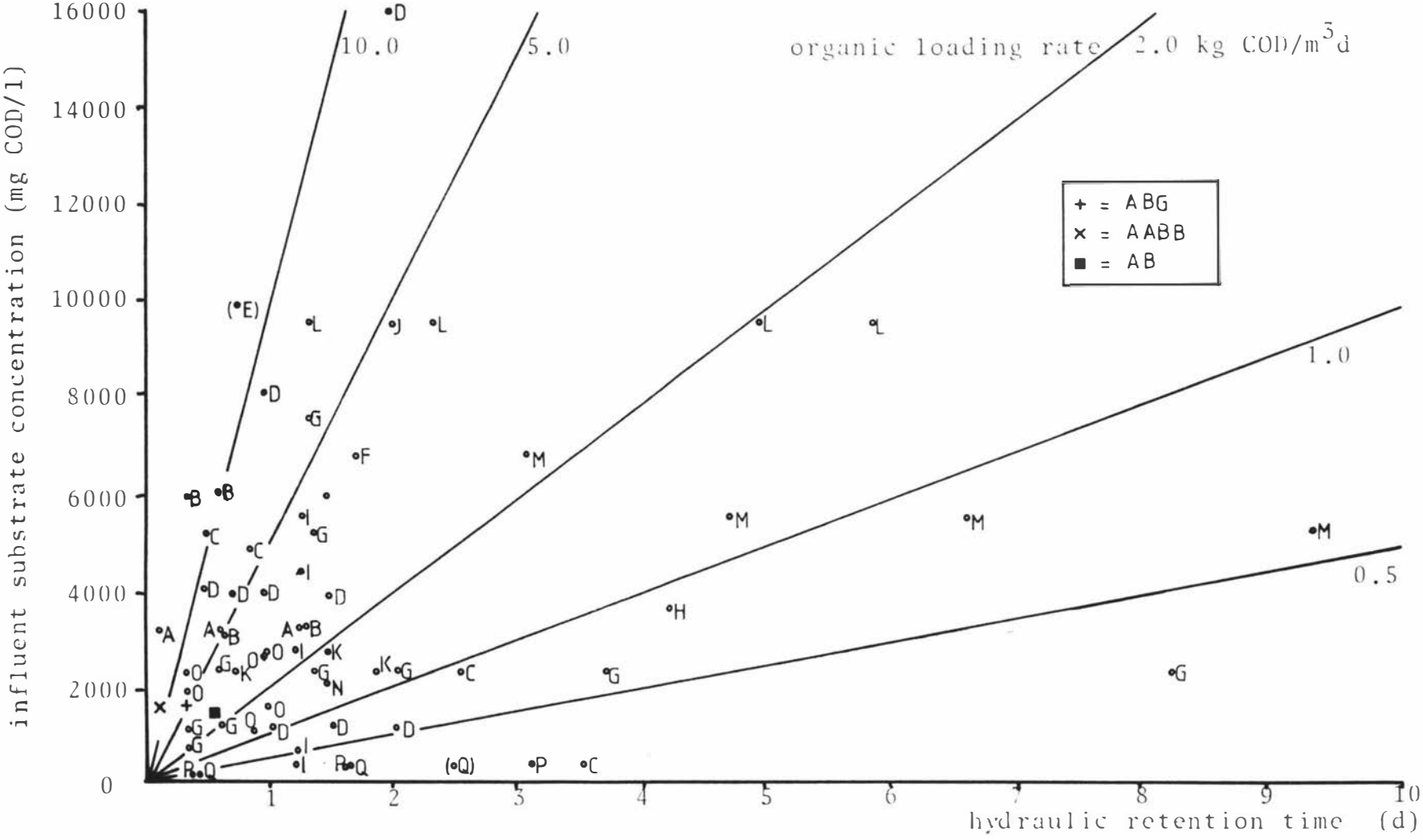


FIGURE 2.6: Influent substrate concentration as a function of hydraulic retention time for data from Table 2.6 with organic loading rates indicated.

The legend for Figure 2.6 is as follows:

A Young and McCarty (1967) protein and carbohydrate waste.

B Ibid - VFA waste.

C Plummer *et al* (1968)

D Jennett & Dennis (1975)

E El-Shafie & Bloodgood (1973)

F Taylor & Burm (1975)

G Peterson (1975)

H Force & Lovan (1971)

I Arora *et al* (1975)

J Haug *et al* (1977)

K Hovious *et al* (1971)

L van Velsen *et al* (1979)

M Norman & Frostell (1977)

N Sachs *et al* (1978)

O Anderson *et al* (1978)

P&Q Hudson *et al* (1978)

In terms of organic loading rate, based on the initial void volume of the filters, the points (E) and (Q) in Figure 2.6 represent respectively the highest loading rate of 18.75 kg COD/m^3d and the lowest of 0.15 kg COD/m^3d. It is axiomatic that any number of combinations of substrate concentration and retention time will yield the same organic loading rate and such are also indicated in Figure 2.6. In terms of this parameter, most studies have been concentrated in the range of 1.0 kg COD/m^3d to 10.0 kg COD/m^3d,

which is in the range generally associated with anaerobic digestion processes.

2.4.3 Removal of Organic Material

The percentage removal of COD by an anaerobic filter is illustrated in relationship to the organic loading rate in Figure 2.7. Although there is much scatter in the data there is a general trend towards a lower percent removal at higher organic loading rates. Regression lines calculated for each data set using the least squares criterion and forcing the intercept through the point 100 percent COD removal at zero organic loading rate have been drawn in on Figure 2.7. The statistical significance of each line is presented in Table 2.7. The data sets omitted from Table 2.7 are so because of insufficient data points to compute the regression line.

Good correlation between percent COD moved and organic loading rate were observed for a protein/carbohydrate waste (Young & McCarty, 1967), and a shellfish processing waste (Hudson *et al*, 1978). Somewhat less well correlated data was observed for the tannery waste (Arora *et al*, 1975), the protein/carbohydrate waste of Norman & Frostell (1977) and the shellfish processing waste water treated with a rock-filled anaerobic filter (Hudson *et al*, 1978). However, the degree of treatment performance seemed not only dependent on the type of waste treated but on the particular system used by each study, as shown by the difference between the protein/carbohydrate waste performances of Young & McCarty (1967) and Norman & Frostell (1977). These had slopes of -1.857 and -3.240 (% COD /kg COD/m^3d) respectively. Conversely there was similari between the performance figures for both the protein/carbohydrate and volatile acid wastes treated on the same filters by Young & McCarty (1967).

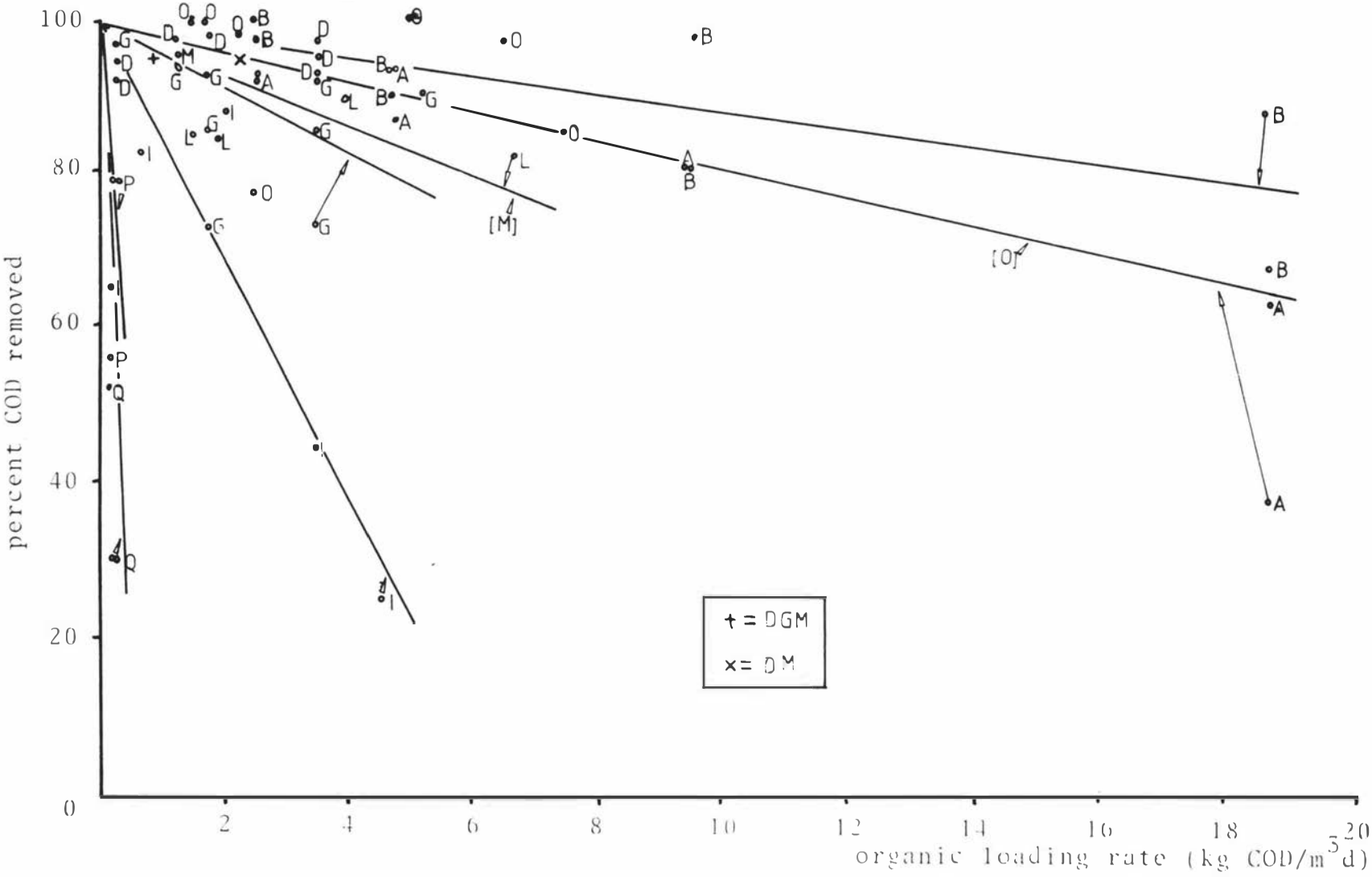


FIGURE 2.7: Percent COD as a function of organic loading rate. Data from Table 2.6.

TABLE 2.7 Details of Regression Lines in Figure 2.7.

Data Set	Slope $(\frac{\%\ COD\ (R)}{kg\ COD/m^3 d})$	Coefficient of determination (r^2)	Waste-type	Source
A	-1.857	0.971	protein/carbohydrate	Young & McCarty (1967)
B	-1.205	0.789	volatile acid	Young & McCarty (1967)
D	-1.225	0.441	pharmaceutical	Jennett & Dennis, (1975)
G	-4.136	0.603	milk	Peterson (1975)
I	-15.37	0.859	tannery	Arora *et al* (1975)
L	-3.22	0.786	calf manure	van Velsen *et al* (1979)
M	-3.240	0.808	protein/carbohydrate	Norman & Frostell (1977)
O	-1.865	0.522	NO_3-N/brewery	Anderson *et al* (1978)
P	-117.6	0.956	Shellfish processing[1]	Hudson *et al* (1978)
Q	-193.1	0.830	Shellfish processing[2]	Hudson *et al* (1978)

[1] oyster shell filter packing

[2] rock filter packing

The available BOD_5 data (Table 2.6) indicated similar trends to COD data (Figure 2.8).

The initial anaerobic filter investigation (Young & McCarty, 1967) was used to develop a mathematical model incorporating biological growth, solids transport, diffusional resistance of the biofilm and non-steady state solids build-up. This model was considered too complex for analysis or design (Mueller & Mancini, 1975). A simple equation to predict the removal of soluble ultimate BOD was also presented (Young & McCarty, 1975)

$$E = 100\ (1-\frac{\varepsilon}{t}) \qquad (2.9)$$

where E = % ultimate BOD removal
t = hydraulic detention time
ε = a system constant

For the system studied, at 25°C the value of ε was found to be 1.8 h. Figure 2.9 is a plot of observed COD removals as a function of hydraulic retention time for anaerobic filter studies (Table 2.6) and superimposed is equation 2.9. This may be seen as a midrange of very scattered data points. Lower removals than those predicted by equation 2.9 were considered to be due to the nature of the waste or to high solids carryover (Mueller & Mancini, 1975).

A kinetic study of the anaerobic filter (Mueller & Mancini, 1975) concluded that a complex model employing Michaelis kinetics, a pH inhibition function and separate organism fractions but neglecting solids transport and biofilm diffusion simulated the steady state data. But a simple model employing first order kinetics approximates the performance and was more readily useable for system design and analysis. It must be noted that the former model was based on an assumption that the organic carbon was converted to methane in two stages by conversion to volatile acids thence to methane, which is now not in favour.

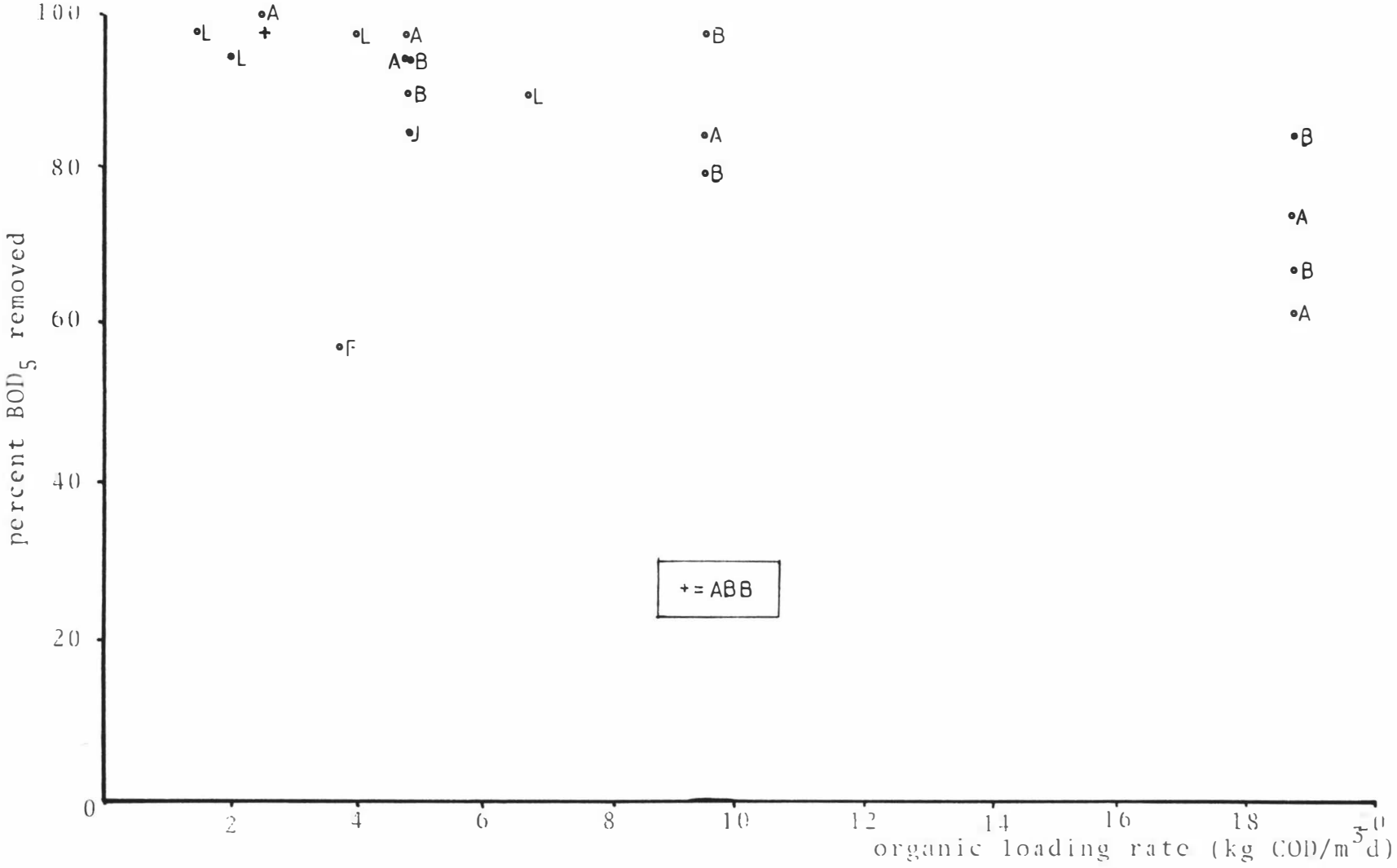


FIGURE 2.8: Percent BOD_5 removed as a function of organic loading rate. Data from Table 2.6.

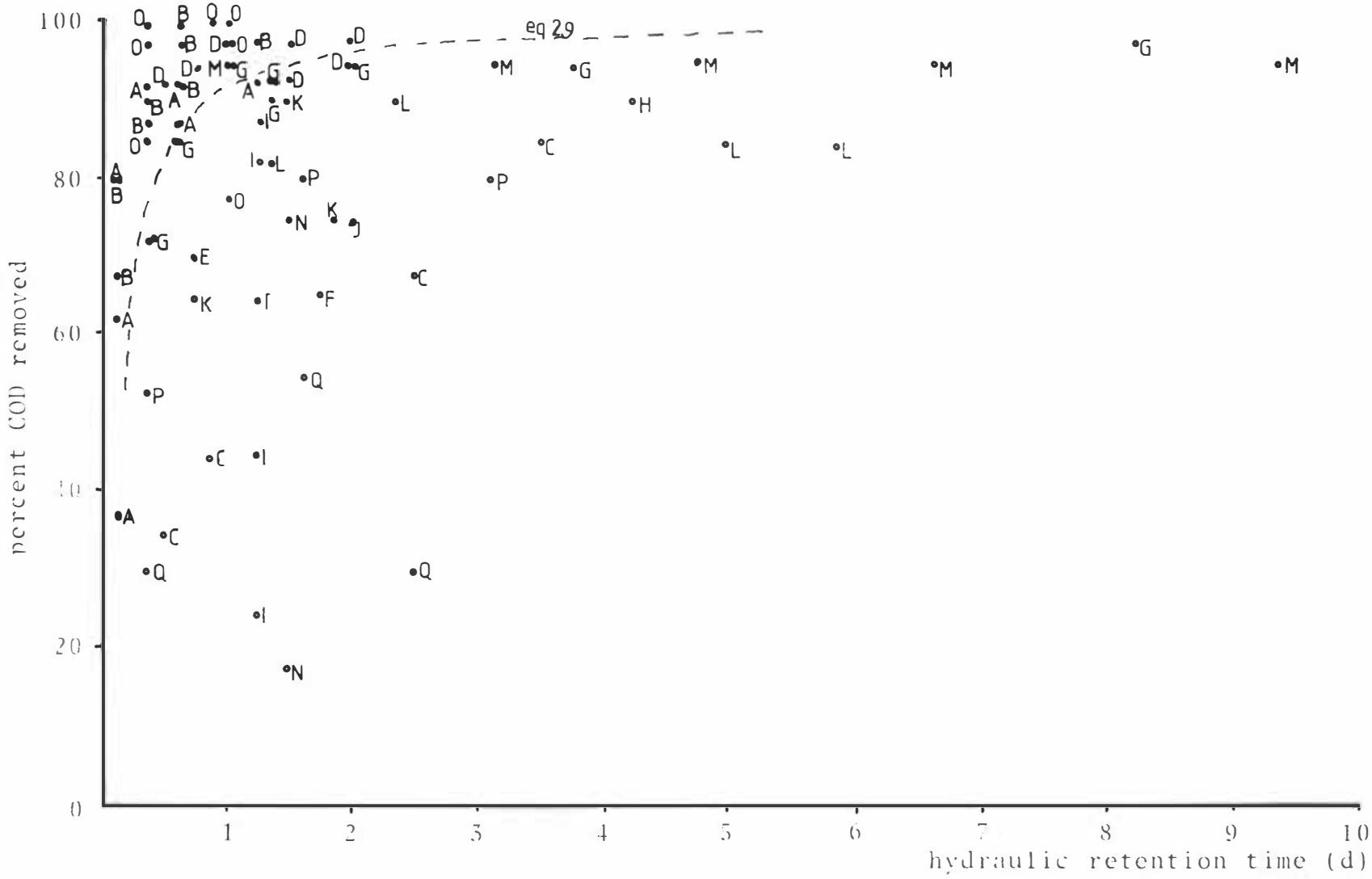


FIGURE 2.9: Percent COD removed as a function of hydraulic retention time. Data from Table 2.6.

The application of first order kinetics to the data in Table 2.6 has been carried out as follows :

From $\ell n \frac{S}{S_o} = -kt$ (2.10)

where S = final substrate concentration (mg/ℓ)
S_o = initial substrate concentration (mg/ℓ)
k = first order rate constant (d^{-1})
t = retention time (d)

it follows that

$$\frac{\ell n \frac{S}{S_o}}{S_o} = -\frac{kt}{S_o} \quad (2.11)$$

and since the organic loading rate is defined as

$$OLR = \frac{S_o}{t} \quad (\frac{mg}{\ell .d}) \quad (2.12)$$

where t is the retention time based on initial void volume,

then from equation 2.11:

$$\frac{S_o}{\ell n \frac{S}{S_o}} = -\frac{OLR}{k} \quad (2.13)$$

and a plot of $\frac{S_o}{\ell n \frac{S}{S_o}}$ against OLR should be linear with slope $-\frac{1}{k}$.

Figure 2.10 is the plot of equation 2.13 for the data in Table 2.6, and Table 2.8 gives values of k calculated from linear regression times.

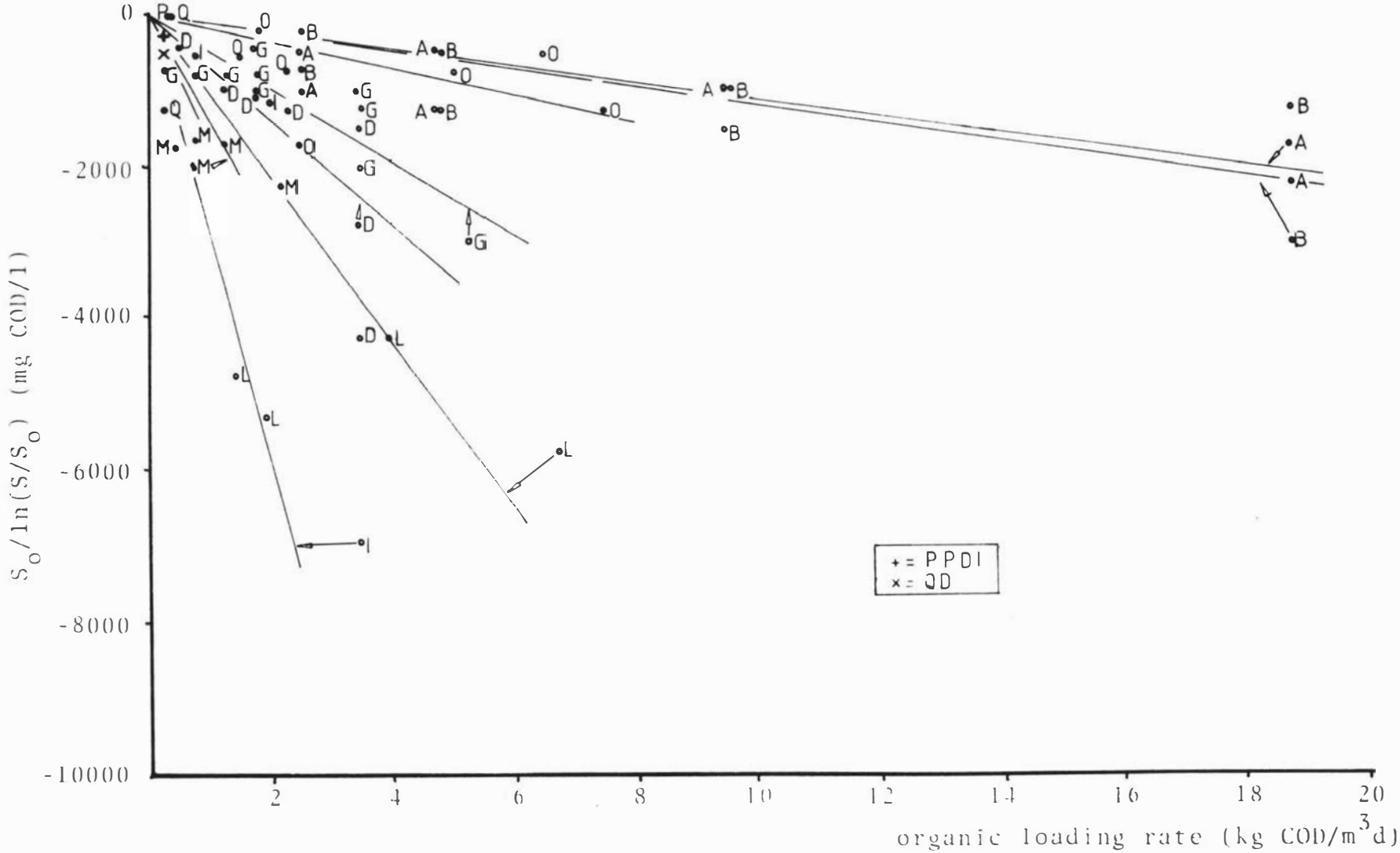


FIGURE 2.10: Plot of $S_o/(\ln(S/S_o))$ against organic loading rate. Data from Table 2.6.

TABLE 2.8: First Order Kinetic Constants for Removal of COD by Anaerobic Filters

Data Set	k (d^{-1}) x 10^6	Coefficient of determination (r^2)	Source
A	8.795	0.878	[1]) Young & McCarty (1967)
B	8.245	0.868	[2]) Young & McCarty (1967)
D	1.45	0.805	Jennett & Dennis (1975)
G	2.09	0.906	Peterson (1975)
I	0.337	0.834	Arora *et al* (1975)
L	0.935	0.778	van Velsen *et al* (1979)
M	0.705	0.842	Norman & Frostell (1977)
O	5.55	0.675	Anderson *et al* (1978)
P	1.58	0.693	3) Hudson *et al* (1979)
Q	0.582	0.399	4)

NOTES

[1] protein/carbohydrate waste
[2] volatile acid waste
[3] oyster shell packing
[4] rock packing

There is a greater range in rates between the different filter systems rather than between waste types as would be expected. For instance the different wastes treated in one study by the same filter (Young & McCarty 1967) showed very similar rate constants for both protein/carbohydrate wastes and a volatile acid one, 8.795 and 8.245 x 10^{-6} d^{-1} respectively. Whereas in another study (Norman & Frostell, 1977) treatment of a protein-carbohydrate waste yielded a rate constant smaller by more than an order of magnitude.

Although the organic loading rate is comprised of both the influent substrate concentration and the hydraulic retention time it is not possible to determine the effects of each parameter from the data in the literature. It has been noted that COD removals are higher at longer hydraulic retention times regardless of the substrate concentration, presumably due to a better balance between the fermentative and methanogenic phases of digestion. Also at longer retention times there will be less washout of free swimming bacteria (Mosey, 1978).

From three studies (Young & McCarty, 1967; Jennett & Dennis, 1975; Peterson, 1975) data at constant organic loading rates were gathered for different hydraulic retention time - influent substrate concentration combinations. The latter two studies noted that, at organic loading rates of 3.52 kg COD/m^3d and at 1.75 and 3.50 kg COD/m^3d, the higher percentage removals of COD were favoured by longer hydraulic retention time and higher influent substrate concentration. Young & McCarty (1967) observed that over a similar range of 2.38 to 4.76 kg COD/m^3d, the percentage COD removed was almost constant although slightly higher for the longer retention time - higher substrate concentration cases. However at high organic loading rates of 9.38 and 18.76 kg COD/m^3d, the percentage COD removed was greatly enhanced by the longer retention time - higher substrate concentration combination.

The effects of hydraulic retention time and influent substrate concentration on effluent COD concentration are illustrated

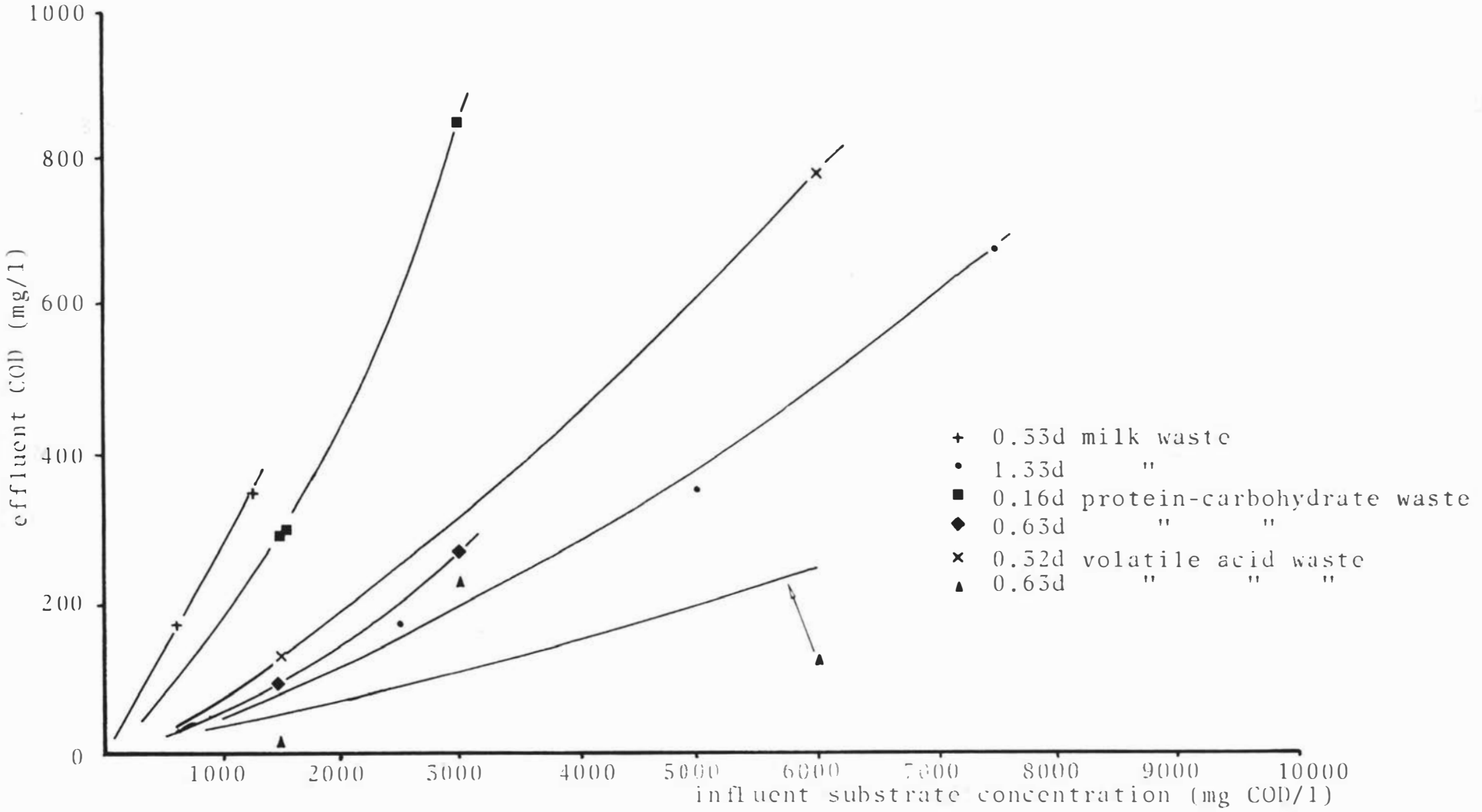


FIGURE 2.11: Anaerobic filter effluent COD as a function of influent substrate concentration. Data from Table 2.6.

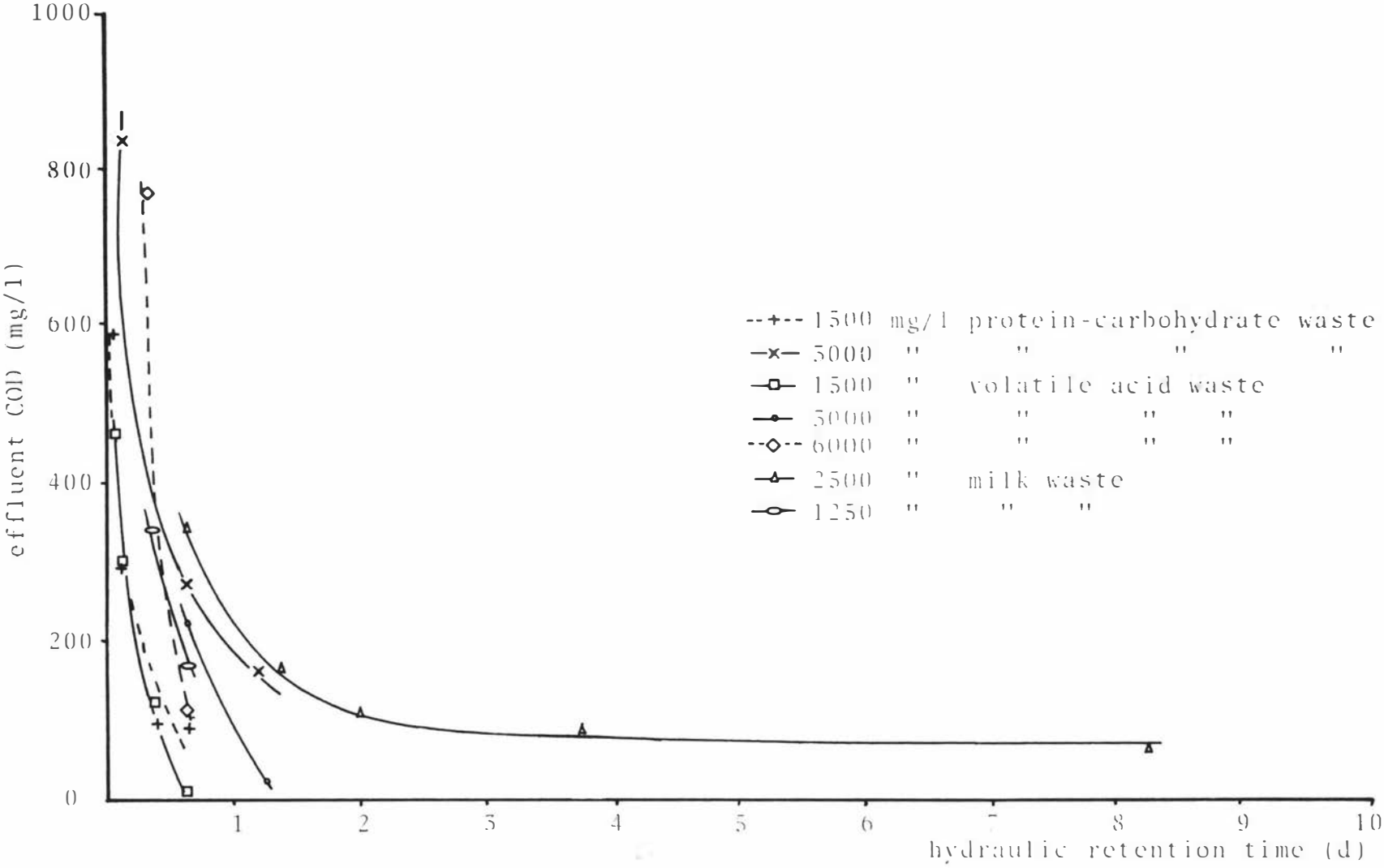


FIGURE 2.12: Anaerobic filter effluent COD as a function of hydraulic retention time for three waste types at different concentrations. Data from Table 2.6.

in Figures 2.11 and 2.12. It was observed that reduced effluent COD resulted from both a reduction in influent COD at constant hydraulic retention time and increased hydraulic retention time at constant influent substrate concentration. Although the magnitude of each effect was not able to be quantified it is clear from the figures that influent substrate concentration was varied over a much greater range of values than the hydraulic retention time.

2.4.4 Conversion of COD to Methane

Table 2.9 is a presentation of the available gas data. Based on the theoretical maximum of 0.35 m^3/kg COD stabilised, column four is the ratio of the actual yield to the theoretical yield. Where more than one figure is available for a given study (Peterson, 1975; van Velsen *et al*, 1979; Norman & Frostell, 1977) the fraction of theoretical yield achieved decreased as the organic loading rate on the system increased. If conversion to methane is also an indication of conversion of COD to cell material (Peterson, 1975), then as the loading rate increased more organic material was being converted to cellular material. This would follow from a consideration of the biochemistry of anaerobic digestion (Bryant, 1979), that as the system become stressed due to a high loading rate the fermentative bacteria would proliferate as conditions became adverse for the methanogenic bacteria. In addition to the data in Table 2.9 several workers have indicated that methane production accounted for 83.5 to 97.9 percent (Young & McCarty, 1967), up to 90 percent (Jennett & Dennis, 1975) and 89 percent (de Walle & Chian, 1976, 1977) of the COD removed; and that the remainder was presumably converted to new cell material.

TABLE 2.9 : Methane Yields for Anaerobic Filtration

Waste type	OLR kg COD/m^3d	Gas production m^3/m^3d	CH_4 content %	Fraction of* theoretical yield	Source
Synthetic ('Metrecal')	13.3	3.41	62	0.60	El-Shafie *et al* (1973)
Milk Processing	0.65	0.421	72	0.79	Peterson (1975)
	1.17	0.414	72	0.78	
	1.75	0.407	70.5	0.75	
	1.75	0.362	70.5	0.67	
	1.75	0.375	70.5	0.69	
	3.5	0.332	70.5	0.61	
	3.5	0.311	70.5	0.57	
	3.5	0.333	69	0.60	
	5.25	0.306	65	0.52	
Heat treated activated sludge	4.75	0.50	70	0.89	Haug *et al* (1977)
Calf manure	1.6	0.618	85.2	0.98	van Velsen *et al* (1979)
	1.9	0.420	87.6	0.60	
	4.0	0.841	86.3	0.58	
	6.7	0.950	86.9	0.39	
Synthetic carbohydrate and protein	0.56			0.57	Norman & Frostell (1977)
	0.68			0.66	
	1.17			0.75	

* corrected for temperature

2.4.5 Recirculation of Filter Effluent

The use of single pass anaerobic filters has generally been the case in most previous studies. For this condition, steady state conditions were assumed for a plug flow regime and kinetic models derived using zero and first order kinetics (Jennings *et al*, 1976) and Monod kinetics (Young & McCarty, 1967).

Variations in the parameters, pH, COD, volatile acid concentration were observed at different column height increments (Young & McCarty, 1967; Jennett & Dennis, 1975; Mueller & Mancini, 1975). To prevent large pH drops at the bottom (inlet) of the filters, due to rapid volatile acid production, large quantities of buffer solutions needed to be added. Further, the majority of the COD removed by the filter was removed in the bottom portion of the filter column.

To prevent such pH swings and utilise the entire column height, a flow regime in which most of the filter effluent was recirculated, was proposed (de Walle & Chian, 1976). The flow pattern ensured that the filter was essentially a completely mixed system. High strength, acidic waste streams could be treated by such a process without the need for the constant addition of alkali. Internal pH was maintained by dilution of the feed by the recirculated effluent which also retained buffer capacity especially in the form of ammonia. Recirculation was also predicted to improve filter effluent quality. This latter was based on previous observations that at constant organic loading rate better effluent quality resulted from a lower influent substrate concentration (Jennett & Dennis, 1975).

The improvement in effluent quality was not demonstrated however and further studies concluded that circulation of the filter effluent did not improve treatment capacity either (Johansen & Carlson, 1976).

On the other hand the benefits of reduced risk of shock loading, toxic material dilution and retention of buffering

capacity have been well demonstrated (Witt *et al*, 1979)

2.4.6 Filter Start Up

In all the studies on the anaerobic filter (AF), the test units have been seeded with material from an actively operating anaerobic digester, although alternative seed sources are available, for instance cattle manure (Sen & Bhaskaran, 1964). Young & McCarty (1967) evaluated two methods of seeding the AF. A heavy seed of volatile solids from an anaerobic digester introduced at the base of the filter initially and again after 20 d operation gave rise to greater than 90 percent COD removal in the AF after 40 d. A lighter seed distributed throughout the column height resulted in the AF achieving 90 percent COD removal after 80 d, thus the heavy bottom seed was recommended for filter start up to prevent washout of the slower growing methanogenic organisms. This advice has been followed by later workers.

Substrate choice at start up is either between a much diluted solution of the ultimate test material or a synthetic substrate and the AF is 'weaned' onto the test material. Both methods have been utilised. Jennett & Dennis (1975) and Sachs *et al* (1978) both used the AF to treat a pharmaceutical waste; the former started their AF on a glucose and trace element mixture and the latter used 2000 mg COD/ℓ methanol solution; the AF was gradually 'weaned' onto pure pharmaceutical waste over a period of some weeks. El-Shafie & Bloodgood (1973), after seeding their multiple effect AF with active anaerobic digester supernatant, started with an AF feed concentration of 1000 mg COD/ℓ which was increased to 10000 mg COD/ℓ over a period of 12 weeks. The AF was started on the substrate used throughout the test.

As far as restarting the AF after a break in operation goes, the filter can be operated without re-seeding after shut down periods of several months (Taylor & Burm,1975; El-Shafie and Bloodgood,1973) which would be advantageous for treatment of waste waters on a seasonal or intermittent basis. Peterson

(1975) noted that much of the biomass in the AF was associated with the interstices in the packing medium and only a thin film was actually attached to the medium itself. To test a method of cleaning the AF, the 'interstitial' biomass was flushed out and after restart at a low OLR (0.65 kg COD/m^3d) a healthy methane production was manifest after 24h operation. A simple backwash for solids removal from the filter was proposed.

2.4.7 Nutrient Requirements

Due to the low level of solids synthesis generally experienced with anaerobic digestion, the requirement for nitrogen and phosphorus nutrients is much lower than for aerobic processes.

To ensure that the filter operation was not limited due to nutrient availability, Jennett & Dennis (1975) added nitrogen and phosphorus to maintain a COD:N and COD:P ratios of 100:5.9 and 100:1 respectively. Taylor and Burm (1975) also added nitrogen to enrich the COD:N ratio of 100:4.2 present in the wheat starch effluent treated by their full-scale AF. In both cases the nutrients were added to the filter with the feed material.

In other studies, low nutrient levels in the feed were not considered to adversely affect AF operation even though the COD:N and COD:P ratios were respectively 100:0.37 and 100:0.22 (Foree & Lovan, 1971) and 100:2.6 and 100:0.023 (de Walle & Chian, 1976), as appreciable quantities of both nitrogen and phosphorus were found in the AF effluent.

CHAPTER THREE

INTRODUCTION TO THE EXPERIMENTAL DESIGN

CHAPTER THREE

INTRODUCTION TO THE EXPERIMENTAL DESIGN

3.1 Statistical Experimental Designs

The classical approach to scientific experimentation in which each factor studied is varied individually while others are held at a constant or base level has several drawbacks. The response of the altered state is compared to a control one in which all factors are held at the base level. This allows for evaluation of the magnitude of the effect of each factor, but one cannot;

(a) attach confidence levels to the estimates of such effects;

(b) estimate experimental or residual error in the test data;

(c) estimate the effects of interaction between two factors.

(Lipson & Sheth, 1973)

This last point has been shown to be more important in biological systems which tend to be far more complex than chemical or physical processes (Maddox & Richert, 1977).

An alternative approach to experimental design is to use a factorial design and analysis of variance techniques. In a factorial experiment several different sources of variation may be acting simultaneously on a set of tests and so the variance of the observations is the sum of the variances of each independent source. Analysis of variance techniques may then be applied to break this total observed variance into variances due to each main factor, interaction between factors and residual or experimental error. Each variation is tested for statistical significance.

Consideration of a hypothetical experimental design at this point would be appropriate to define some terms. The

response of this system is considered to be subject to the effects of independent parameters, say A,B and C. Each of these parameters is to be tested at two levels. The full factorial experiment for this case is defined in Table 3.1.

TABLE 3.1 : Experimental Design For a Full Factorial Experiment Involving Three Factors at Two Levels Each

	Factor			
Run	A	B	C	Code
1	-1	-1	-1	-1
2	1	-1	-1	a
3	-1	1	-1	b
4	1	1	-1	ab
5	-1	-1	1	c
6	1	-1	1	ac
7	-1	1	1	bc
8	1	1	1	abc

-1 indicates a factor at its low level
1 indicates a factor at its high level

A factor is a process variable which can be maintained at a fixed level. The factors are also known as the independent invariables and the experimental designs are based on the combinations of these variables. Factors may be either quantitative, for instance a concentration; or qualitative, such as the presence or absence of a particular species.

An experimental run or trial is one set of combinations of factor levels defined in the experimental design. In Table 3.1 each row represents one experimental run.

The response of the system under study is any measured parameter from an experimental run. Any number of response variables may be used in the significance testing of the

factors. System response variables are termed the dependent variables.

In a factorial experiment although several of the independent variables are varied simultaneously, variables other than those under investigation must be strictly controlled. To minimise the effects of operator and environmental influences, the order in which the runs are carried out is randomised. Since analysis of variance depends on the laws of probability, such randomisation should distribute the effects of unknown or uncontrollable influences.

Analysis of the data from factorial experiments to ascertain the significant variables and interactions is well illustrated in statistical texts (Lipson & Sheth, 1973; Chatfield, 1970). Computations of analysis of variance are relatively simple for two and three factor experiments but become progressively more difficult as the number of factors are increased.

3.2 System Modelling

Experimental designs lend themselves well to mathematical modelling using regression analysis. Regression analysis is used to determine the functional relationship between variables. This is distinguished from correlation analysis which determines the degree of association between variables. Regression models are quantitative relationships between the selected experimental factors and the response variable and are used for process description, prediction of process behaviour and process control and optimisation.

Such models have the form

$$y = f(x, \beta) + e \qquad (3.1)$$

where y = the dependent variable

x = the independent variables ($x, \ldots x_k$)

β = regression parameters or coefficients of the independent variables determined from the

sample data

e = uncertainty in the model

f = the response function which describes a surface in k dimensional space.

The regression equation is a response surface and the standard regression model is linear in the regression parameters although the process variable levels may be the result of complex non linear functions.

Models of systems may be either empirical, such as regression relationships or mechanistic. The latter are based on underlying physical or chemical considerations and give an insight into the process mechanism. The parameters in mechanistic models have physical significance, are usually complex, and the models can be extropalated beyond the range of data used to elucidate them.

Empirical models on the other hand have parameters of no particular physical significance and cannot safely be extrapolated beyond the limits of the experimental range.

There are different classes of experimental design and they find application depending on the type of result sought. Box (1964) describes an on going process of design and analysis (Figure 3.1) utilising the different classes of experiment. In Figure 3.1 the initial design would be a simple screening test to find the significant variables, this would be followed by designs to elucidate models with subsequently higher orders of parameter estimates.

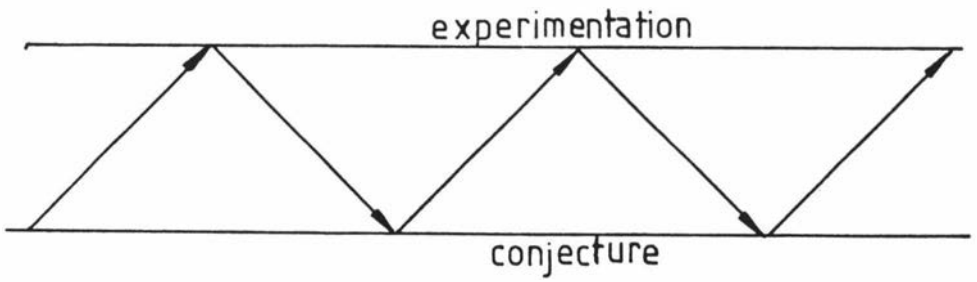


FIGURE 3.1 : The model building process (Box, 1964)

Using this procedure a small sub-region of the response surface is finally delineated which may be expected to contain an optimum set of conditions. An example of this method is used by Ramsay (1982).

After deciding upon the design and carrying it out to collect the data the analysis to estimate factor effects and regression equations is described in the following section

3.3 Types of Experimental Design

There are many types of experimental design. However, for the purposes of this study only a brief mention of two classes of design will be made. These are the first and second order factorial designs. The order of these designs refers to the order of the polynomial generated from regression analysis of the design. *First order models* have factors tested at two levels each and this approximation assumes the response surface is planar. A two factor, first order response surface is shown in Figure 3.2

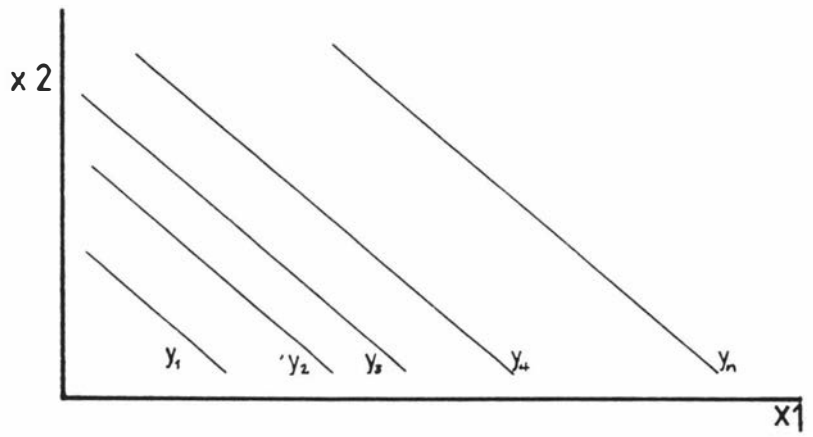


FIGURE 3.2 : Response surface for a general two factor first order model

The generalised first order response equation is defined in equation 3.2.

$$Y = \beta_o + \beta_i x_i + \beta_{ij} x_i x_j + \beta_{ijk} x_i x_j x_k + \bar{e} \qquad (3.2)$$

where β_o = a constant

β_i = a coefficient of the main effect of factor x_i

β_{ij} = a coefficient of the interaction between factors x_i and x_j

β_{ijk} = a coefficient of the interaction between factors x_i x_j and x_k.

e = experimental error

Regression techniques are used in the analysis of experimental designs and whereas the true value of parameter, β_i, will always be unknown, statistically significant estimates of the parameters may be made. To indicate that these factor parameters are estimates the notation $\hat{\beta}_i$ is used.

In most modelling situations the interaction of greater than two main effects is ignored as three factor or greater interactions are considered to be extremely rare.

A full factorial design for a first order design having k factors has 2^k runs. The resulting model fits the data exactly at the experimental points but no information is available about lack of fit or pure error. The aim of these experiments is usually to screen a number of variables to identify those most significant and to test for interactions.

Full 2^k experimental designs require many runs. For instance, a 6 factor design requires 64 runs for the full design without any replication, and generates sufficient degrees of freedom to evaluate a grand mean β_o, six main effects, 15 two factor interactions, 20 three factor interactions, 15 four factor interactions, 6 five factor interactions and one six factor interaction. Holding to the assumption that interactions of greater than two main effects are to be ignored then much of the previously mentioned information is wasted. To economise, the experimenter may elect to perform only a fraction of the full design such as ½ or ¼. This practise however leads to confounding of effects in each other. In a half replicate experiment, each column in the $\underline{x}$ matrix has a twin in the part of the design not carried out and in this case the coefficients are estimated in pairs.

Fractional factorial designs have defining relationships which indicate which parameters are confounded in each other. For example a design with the defining relationship :

$$D = AB = CG = EF \tag{3.3}$$

means that the estimate of the main effect of factor D is actually the sum of the effect of D plus the interactions AB, CG and EF, or :

$$\hat{\beta}_D = \beta_D + \beta_{AB} + \beta_{CG} + \beta_{EF} \tag{3.4}$$

and positive main effects may therefore be cancelled out by several negative interactions. To minimise the latter possibility, the fraction of the full design is chosen which has the desired parameters confounded with high order interactions which are unlikely to be significant. Tables of fractional factorial designs are available in the literature for given main effects and interactions for up to ten factors (Davies, 1954; National Bureau of Standards, 1957 and 1959).

Second Order Designs

If a large lack of fit has been found from a first order model or if an optimum is suspected or a significant deviation from linearity has been found, the experimenter may choose a second order experimental design.

Second order models have the general form of equation 3.5, in which the interactions of greater than two factors and interaction between linear and quadratic terms are considered to be optional

$$Y = \beta_o + \beta_i x_i + \beta_{ij} x_i x_j + \beta_{ii} x_i^2 + \beta_{ijk} x_i x_j x_k + \beta_{ijj} x_i x_j^2 + \beta_{iijj} x_i^2 x_j^2 \tag{3.5}$$

The two types of second order designs are composite designs, which are two level factorials with additional runs at mid range and/or outlying points and three level factorial or fractional factorial designs. The former are not suitable for use with qualitative factors as up to five levels of each factor are utilised.

Factorials of 3^k runs are expensive of time and materials if k>3, but they can be fractionated, although not as simply as for two level designs. For instance there are 24 different 3^{4-1} designs in which the defining contrast is a four factor interaction.

The nomenclature 3^{4-1} is used as opposed to 3^3 to indicate that the experiment is a fraction of a full four factor experiment not a full 3 factor one. In addition to true fractional factorial designs various incomplete designs are available for three level and mixed two and three level designs (Webb, 1971).

3.4 Data Analysis

It has been noted previously that analysis of variance is a powerful technique in statistics. For a simple one factor experiment, a one way analysis of variance may be used to analyse the data. Two factor experiments may be tested for significance using a two way analysis of variance or the algorithmn of Yates. Regression modelling involves fitting a curve to the observed data. The criterion adopted is that of least squares.

To estimate the magnitude of the effects of greater than two factors, matrix notation is utilised which readily lends the data to manipulation by digital computors. The matrix representation for the model

$$\underline{Y} = \underline{\beta}\ \underline{x} + \underline{e} \qquad (3.6)$$

where the underscored parameters are matrices is as follows

$$\underline{y} = \begin{bmatrix} y_1 \\ y_2 \\ \cdot \\ \cdot \\ y_n \end{bmatrix} \qquad \underline{x} = \begin{bmatrix} 1 & x_{11} & x_{21} \cdots\cdots x_{p1} \\ 1 & x_{12} & x_{22} \cdots\cdots \\ \cdot & \cdot & \cdot \\ \cdot & \cdot & \cdot \\ 1 & x_{1n} & \cdot \quad \cdots\cdots \quad x_{pn} \end{bmatrix}$$

$$\underline{\beta} = \begin{bmatrix} \beta_0 \\ \beta_1 \\ \cdot \\ \cdot \\ \beta_n \end{bmatrix} \qquad \underline{e} = \begin{bmatrix} e_1 \\ e_2 \\ \cdot \\ \cdot \\ e_n \end{bmatrix}$$

This model is for experimental design involving p factors and n runs or observations. Estimation of the regression parameters $\hat{\underline{\beta}}$ is carried out by multiple linear regression using the least squares criterion. In matrix form the least squares estimate is

$$\underline{x}^T \underline{x} \hat{\beta} = \underline{x}^T \underline{y} \tag{3.7}$$

and solving for $\hat{\underline{\beta}}$

$$\hat{\underline{\beta}} = (\underline{x}^T \underline{x})^{-1} \underline{x}^T \underline{y} \tag{3.8}$$

In equations 3.7 and 3.8, $\hat{\underline{\beta}}$ indicates the matrix in an estimate of the true $\underline{\beta}$. The superscripts T and -1 indicate the transpose and inverses of the relevant matrices respectively. Calculating the matrix of coefficient estimates is simplified by transposing the levels of the independent variables into a coded form. For two level factors the higher level is

represented as the value 1 and the lower level as -1. Each element in the matrix $\underline{x}$ is transposed as follows :

$$x_{ij} = \frac{\text{actual value} - \frac{1}{2}(\text{High value-low value})}{\frac{1}{2}(\text{high value-low value})} \quad (3.9)$$

Three level factors are coded as -1, 0 and 1 for the low, mid range and high levels respectively. This coding system assumes that smaller numerical values of the factor levels do not lose significance to the larger ones in the calculation of parameter estimates. The levels for a three level factor must also be equally, but not necessarily linearly spaced. They may for instance have equally spaced logarithmns or indices.

The matrix of residuals, $\underline{e}$ is calculated from the fitted model using equation 3.10.

$$\underline{e} = \underline{y} - \hat{\underline{y}} \quad (3.10)$$

The residuals are the differences between the actual system response $\underline{y}$ and the responses predicted by the model, $\hat{\underline{y}}$. Residuals arise from random variation and lack of fit in the model. Inspection of the residual matrix is important as large deviations from predicted behaviour could indicate mistakes in measurements and influence the validity of assumptions made. The residuals are also used in the estimation of the variance of the parameter estimates using equations 3.11 and 3.12 in which s^2 is the estimate of σ^2.

$$s^2 = \underline{e}^T \underline{e}/n-p \quad (3.11)$$

The variance, co-variance matrix $(\underline{x}^T \underline{x})^{-1} s^2$, provides information of the interdependence of the parameters as follows :

$$\underline{c} = \begin{bmatrix} c_{11} & c_{21} & \cdot & \cdot & \cdot & \cdot & \cdot & c_{p1} \\ c_{12} & \cdot & & & & & & \\ \cdot & & & & & & & \\ \cdot & & & & & & & \\ \cdot & & & & & & & \\ \cdot & & & & & & & \\ c_{1p} & \cdot & \cdot & \cdot & \cdot & \cdot & \cdot & c_{pp} \end{bmatrix} \qquad (3.12)$$

where c_i = the variance of $\hat{\beta}_i$

c_{ij} = the co-variance of $\hat{\beta}_i$ and $\hat{\beta}_j$

The correlation of $\hat{\beta}_i$ and $\hat{\beta}_j$ is given by

$$\frac{\text{co-variance } (\hat{\beta}_i, \hat{\beta}_j)}{(\text{var. } \beta_i \text{ var. } \beta_j)^{\frac{1}{2}}} = \frac{C_{ij}}{(C_i C_j)^{\frac{1}{2}}} \qquad (3.13)$$

A significantly high correlation indicates that the estimated value of one parameter is very much dependent on the chosen value of another and results from the chosen design of experiment. If the removal of one or more factors in the design leaves the other parameter estimates unchanged the design is said to be orthogonal. This situation may only arise if the $\underline{x}^T \underline{x}$ matrix is diagonal. If $\underline{x}^T \underline{x}$ is diagonal then the variance-co-variance matrix will also be diagonal (equation 3.12) and the correlation between $\hat{\beta}_i$ and $\hat{\beta}_j$ must be zero (equation 3.13).

While such independence of parameter estimates of each other is desirable in the data analysis, it is by no means essential. Since orthogonality is dependent on the design of the experiment, where a choice of designs is possible the most orthogonal is usually chosen. The magnitude of the determinants of $\underline{x}^T \underline{x}$ for each design are evaluated and

the larger the magnitude, the better the orthogonality.

The statistical significance of the terms in the regression equation is assessed using the F and t test statistics. A simple and parsimonious model is desirable from both statistical and practical points of view. Statistically, the variance of the prediction increases with the number of terms and just as a model with too few terms underfits the data, one with too many overfits it. In the analysis of variance, parameters which are not statistically significant are deleted. This in effect means that the null hypothesis implicit in F and t test statistics is accepted.

To see how well the estimated regression line fits the observations from the experimental design, a quantity known as the coefficient of determination is found. The total variation of y about the regression line is partitioned in the analysis of variance into the explained and unexplained components represented respectively as the sum of squares due to regression and the residual sum of squares. The coefficient of determination is the ratio of explained variation of total variation and this measures how well the model fits the data. The closer the value of the coefficient of determination is to unity the better the model fits the data. When the regression equation describes a straight line the coefficient of determination is the square of the correlation coefficient.

The choice of the number of terms included in the parsimonious model is up to the experimenter. Obviously all statistically significant terms will be included but some terms may also be included which, although only marginally significant, improve the fit of the model as measured by the coefficient of determination.

CHAPTER FOUR

EXPERIMENTAL PROGRAMME

CHAPTER FOUR

EXPERIMENTAL PROGRAMME

4.1 Scope of This Study

It has been shown in an earlier chapter that

- The anaerobic filter is capable of treating waste water streams, whose organic material is essentially in a dissolved state, with shorter retention times than those required for conventional anaerobic processes.
- Yeast plant effluent is a waste water stream, the organic component of which is predominantly dissolved, and this waste stream is amenable to anaerobic waste treatment.
- Statistically based experimental designs may be more efficient at generating data than the classical, one change at a time, method.

Based on the above observations this study was initiated to generate design relationships for the anaerobic filtration of a high strength liquid waste namely yeast plant effluent. Such design data are lacking in this field.

It was envisaged that such relationships could be used to design an on-site waste treatment system.

By using a statistically based experimental design, empirical relationships between system response parameters and several independent process variables can be generated using computerised linear regression techniques.

In the following section, the selection of variables to be tested and the experimental procedure are discussed.

4.2 Experimental Procedure

4.2.1 Selection of Variables to be Tested

Selection of the independent process variables must of necessity be careful since the number of experimental runs required is related directly to the number of variables to be tested. The variables chosen also had to be those thought to be of major significance from an engineering design point of view. From a review of current literature, five parameters were chosen initially. These were:

A Influent substrate concentration (ISC)
B Hydraulic retention time (HRT)
C Temperature
D Recirculation ratio
E Anaerobic filter size

Factors A and B above can be combined to generate the parameter known as organic loading rate and are of importance in any microbial fermentation. By varying ISC and HRT independently of each other, a range of organic loading rates is possible and two different combinations of these could yield the same organic loading rate.

Temperature was included in the independent variables because all biological reaction rates are dependent on temperature. Further, the temperature at which the yeast plant effluent leaves the propagation vessel is approximately 30^{o}C which is below that considered to be optimal for mesophilic anaerobic digestion of 35 to 37^{o}C.

The relatively high concentration of fermentable sugars in the waste stream leads to its ready conversion to organic acids by fermentative bacteria. To avoid consequent reduction in pH some recirculation of the filter contents was considered desirable. Such recirculation would even out potentially damaging pH fluctuation throughout the filter height and effect a more or less mixed system. The amount of recirculation was chosen as an independent variable to

ascertain its effect on process efficacy.

Finally, since two filter units were to be used in the experimental program and these differed in size, this difference had to be considered as a qualitative independent variable. An indication of the effect of anaerobic filter scale may be gathered from the magnitude of the effect of this variable since the large filter was approximately three times the net volume of the smaller.

4.2.2 Selection of the Experimental Design

In order to detect any curvature in the response surfaces generated from the experimental data it was necessary to choose a second order experimental design. However, since most three level designs involve many more experimental runs than were justifiable economically within the available time, a full three level factorial design was not possible as this would require 81 runs. In the case of anaerobic digestion studies a period of weeks must be allowed per run for the system to settle at a new set of conditions. This imposes a major constraint on the number of experimental runs which can be carried out.

In consequence a mixed level factorial design was chosen with the factors of influent substrate concentration and hydraulic retention time tested at three levels each and temperature, recirculation ratio and filter size each at two levels. Additionally instead of the full replicate $3^{2} \cdot 2^{3} = 72$ run design, a half replicate fractional factorial, 36 run design was chosen.

However, this design was reviewed after the first four experimental runs when it was realised that insufficient time would be available to complete the design and allow further trials to verify the experimental models generated.

In the design completed the factor relating to effluent recirculation ratio was dropped and an effluent recirculation to feed ratio of 4 was used throughout the trials. The design chosen was an incomplete $3^{2} \cdot 2^{2}$ factorial in 12

runs after Webb (1971) and is shown in Table 4.1. This design was able to retain two of the former runs already completed.

TABLE 4.1 : Experimental Design

Run	Factor			
	ISC	HRT	TEMP.	'AF'
1	-1	-1	-1	-1
2	-1	-1	-1	1
3	-1	0	1	1
4	-1	1	1	-1
5	0	-1	1	1
6	0	0	-1	-1
7	0	0	1	1
8	0	1	-1	-1
9	1	-1	1	-1
10	1	0	-1	-1
11	1	1	-1	1
12	1	1	1	1

In Table 4.1 the values listed for each independent variable (factor) are the coded ones used in subsequent linear regression analysis and are as follows:

1 indicates a factor at the high level
0 indicates a factor at the mid point level
-1 indicates a factor at the low level

Optimisation studies on the response surface equations were carried out by the computer technique of Richert (1972).

4.2.3 Selection of the Factor Levels

The following basis was used in the selection of levels for the experimental factors.

A. Influent substrate concentration. A nominal BOD_5 concentration was chosen as the level of ISC. Since the BOD_5 of the spent yeast fermentation liquor combined with subsequent yeast cream wash waters was approximately 5000 mg/ℓ, this value was chosen as the mid point of the three level ISC factor. Also, 1000 mg BOD_5/ℓ was an approximate concentration of yeast wash water alone and was selected as the low level ISC. To maintain the mid point the high level for this factor was then 9000 mg BOD/ℓ.

B. Hydraulic retention time. A previous study (Stander *et al*, 1971) had shown yeast plant effluent to be amenable to anaerobic digestion in four to six days and since this study sought lower HRT by maintaining a fixed biomass, an HRT of five days was chosen as the high level for this factor. The mid- and low levels were chosen as three and one days respectively since the combination of 9000 mg BOD_5/ℓ ISC and 1 d HRT would give rise to an organic loading rate of 9.0 kg BOD_5/m^3d which is a higher loading rate than previously tested on the anaerobic filter system. Further, two of the ISC and HRT combinations, namely 1000 mg BOD_5/ℓ and 1 d HRT and 5000 mg BOD_5/ℓ and 5 d HRT give rise to the same organic loading rate of 1.0 kg BOD_5/m^3d, thus allowing discrimination between three process parameters; ISC, HRT and organic loading rate.

C. Temperature. The temperature of the spent yeast fermentation liquor is approximately 30°C and this temperature was chosen as the low level for the temperature factor. The high level was set at 35°C which corresponds to an optimum temperature for mesophilic anaerobic digestion.

D. Anaerobic Filter Size. This factor was designated by the availability of materials for construction of the filters. Filter void volumes on which basis the hydraulic retention times were calculated were 2.72 ℓ

and 8.0ℓ for the high and low levels respectively.

4.2.4 Response Variables

Any measured response of the system subject to the effects of the independent process parameters may be designated a dependent process parameter. Those measured in this study are the ones of interest from an engineering point of view in waste treatment technology and are listed in Table 4.2

TABLE 4.2 : Experimental Scope of this Study

Independent Process Parameters	Levels High	Mid.	Low
Influent substrate concentration ISC (mg BOD_5/ℓ)	9000	5000	1000
Hydraulic retention time HRT (d.)	5	3	1
Temperature (^oC)	35	-	30
Filter size (ℓ)	8.0	-	2.72

Dependent Process Parameters Measured

soluble COD removal (percent, kg/m^3d)
pH
gas production (ℓ/d and ℓgas/ℓ net filter volume d.)
percent methane in gas
volatile acids
ammonia
total sulphide
alkalinity
conversion of COD to methane

NOTE

Organic loading rates for the combinations of ISC and HRT tested (kg BOD_5/m^3d)

		ISC (mg BOD_5/ℓ) 9000	5000	1000
	5	1.8	1.0	0.2
HRT (d)	3	3.0	1.67	0.33
	1	9.0	5.0	1.0

4.3 Apparatus

Two bench scale anaerobic filters were constructed for the study. They were based on a design by Jennett & Dennis (1975), and consisted of vertical columns of QVF glass pipe section (J.A. Jobling & Co. Ltd, England) packed with glass Raschig rings. The materials of construction were chosen due to their availability at this time. Figure 4.1 illustrates the filter units. The ends of the column sections were arranged to accommodate lines for influent and effluent at the bottom and top respectively. On the smaller unit (AF1) the filter ends comprised QVF pipe reducers (J.A. Jobling & Co. Ltd, Catalogue reference PR 3/1) with rubber stoppers in the smaller end drilled to accept the feed and effluent lines. On AF2, perspex end plates were constructed which sealed the column ends and were drilled and tapped to accept nylon 'Serkit' connectors (Auckland Tool & Gauge Co. Ltd, N.Z.) to which the feed and effluent lines were attached.

Inside the base of each filter unit a perspex dispersion plate, drilled with concentric 3 mm holes, was fitted to distribute the incoming feed material evenly across the filter cross section.

Both filters were filled with glass 'Raschig' rings 10mm in length. The diameter for the glass tubing used was 6mm for AF1 and 12mm for AF2. To prevent short circuiting of fluid up the column walls plastic annular baffles were placed in the filters as indicated in Figure 4.1. Pumping of the feed material and filter recirculation flows was by peristaltic pumps (Cole-Parmer Instrument Company, Masterflex model 7010) with continuously variable speed controllers. Each filter unit had one pump drive fitted with two pumping heads: a model 7013 head for feed pumping and a model 7014-20 head for effluent recirculation. Silicone rubber tubing was used in the pump impellers and each tubing section provided 5 to 10 days service.

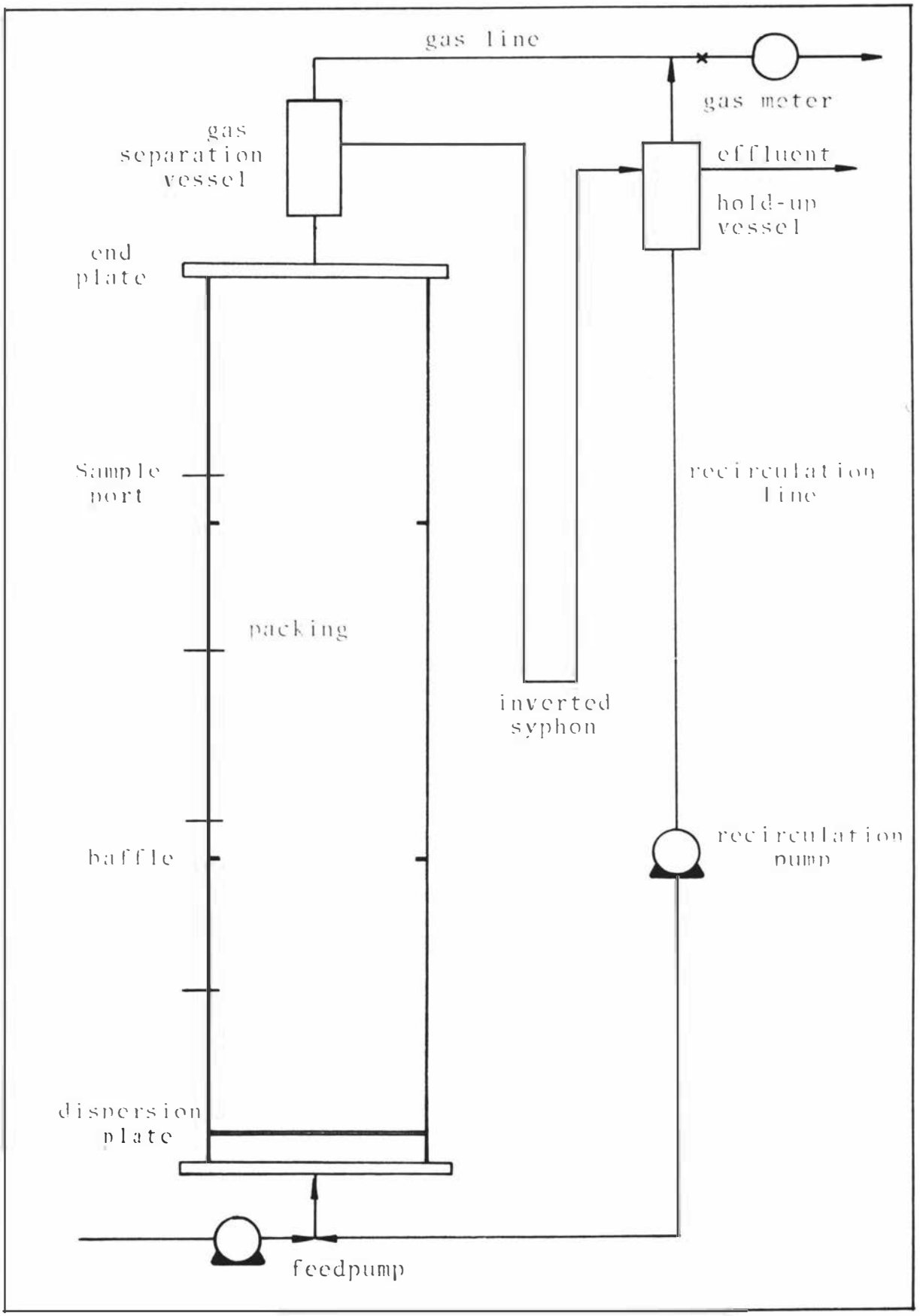


FIGURE 4.1: Schematic diagram of filters used in the experimental trials

To prevent the column from emptying if rupture of the pump line should occur, the pumps were placed above the level of the filter contents, however the difference in levels between the feed reservoir and the filter effluent level provided a positive pumping head.

On the effluent side of the filters an inverted siphon prevented access of air to the filter contents.

Yeast plant effluent diluted with tap water to the required strength was placed in a glass storage reservoir with a magnetic stirrer and renewed daily.

To maintain the anaerobic filters at the required temperature, the entire apparatus was maintained in a walk-in, controlled temperature room. This room was heated by a radiant electric heater and controlled via a temperature controller with an accuracy of $\pm$ 0.5^{o}C. When one of the filters was required to be operated at a higher temperature, a polystyrene box was used to enclose the filter and separate heating connected to the box by two 100 watt light bulbs.

All liquid lines to and from the filter units were 3.2mm PVC tubing which was cleaned of biomass accumulation weekly and replaced as necessary. Lines carrying the digestor gas to the gas meters and then to waste were butyl rubber to prevent loss of methane.

4.4 Sampling Procedures

The two filter units were seeded with an active biomass as described in Chapter 5. Incremental, then continuous feeding of diluted yeast plant effluent was started and gas production and effluent COD monitored as the substrate concentration was increased until the required level was achieved for the first experimental run.

For each experimental run the system was deemed to have achieved a steady state condition when successive gas

production and effluent COD measurements were within ten percent of each other over at least three hydraulic retention times.

Samples were taken for analysis as follows :

Influent substrate concentration - sampled at the feed reservoir for soluble COD.

Effluent from the filters sampled at the inverted siphon for soluble COD, volatile acids, alkalinity, ammonia, soluble sulphide and suspended solids.

Filter internal contents (AF2 only) sampled at ports along the column length in order top through bottom for soluble COD, volatile acids, ammonia, soluble sulphide.

Gas production; daily readings from integrating wet test gas meters attached to the top of the filter units.

Gas composition aliquots of digester gases were taken by syringe from a septum placed on the line taking the gas from the filter units to the gas meters.

4.5 Analytical Techniques

4.5.1 Chemical Oxygen Demand

The COD of both substrate and filter effluent samples were measured by the micro technique developed by Jirka & Carter (1975). The reagents added to 2ml of sample, diluted where necessary to the range 0 to 700 mg COD/ℓ, were : 3ml acid-catalyst solution from a batch of reagent prepared by dissolving 25g silver chloride (AR grade, BDH Chemicals, England) in 2500 ml of water and 1ml 0.250 N potassium dichromate solution prepared by dissolving AR grade potassium dichromate in distilled water. Prior to dilution the sample was centrifuged at 5000 rpm for 15 minutes on MSE Super Minor centrifuge to

remove suspended material.

Digestion of the samples was at 160°C for two hours in screw capped tubes. After this the appearance of Cr(III) was measured spectrophotometrically at 600nm using a Hitachi model 101 spectrophotometer zeroed on a distilled water blank carried through to analysis with the samples.

COD concentration was read from a standard curve prepared using six acid phthalate samples at known COD concentrations of 50, 100, 200, 400, 600, 750 mg COD/ℓ. The standard curve had a regression equation of

COD (mg/ℓ) = absorbance at 600nm x 3270 with a correlation coefficient of 0.999.

4.5.2 5-day Biochemical Oxygen Demand was measured manometrically using HACH model 2173 BOD apparatus (Hach Chemical Co. Ltd, Iowa, U.S.A.).

4.5.3. pH

The pH of all samples was measured, within five minutes of sampling, using an EIL model 7055 pH meter (Electronic Instruments Ltd, Surrey, England). Calibration of the pH meter was by way of buffer solutions at pH 7.00 and 4.00 (EIL, England).

4.5.4 Suspended Solids

Effluent suspended solids and volatile suspended solids were measured by the glass fibre filter paper technique. (Standard Methods for the Examination of Water and Waste Water, 13th Ed., (1975), 208 C). An appropriate aliquot was filtered through a filter disc 2.5 cm in diameter (Whatman Ltd, Maidstone, U.K.) and the disc dried at 103°C for two hours, cooled in a dessicator and reweighed. For volatile suspended solids, portion(s) of this disc and solids were placed in a muffle furnace and ignited at

550°C for 15 minutes and weighed. Blank discs were treated in a like manner and used to correct for moisture and filter disc volatility.

4.5.5 Ammonia

Ammonia in the centrifuged samples was measured using an EIL model 8002-8 ammonia probe connected to an EIL model 7055 pH/concentration meter. The meter was calibrated using known standards of 1.0, 10 and 100 mM ammonium chloride (Analar grade, BDH Chemicals). The samples and standards were adjusted to pH 10 using 1M sodium hydroxide solution prior to testing with the probe.

4.5.6 Sulphide

Total sulphide concentration in the filter contents and effluent samples was measured using an Orion model 94-16 sulphide specific ion electrode (Orion Research Inc., Mass. U.S.A.). A direct measurement technique was used by calibrating the EIL 7055 pH/concentration meter against known standard solutions prepared using $Na_2S.9H_2O$ (Analar grade, BDH Chemicals).

4.5.7 Alkalinity

Alkalinity was measured on all samples by a potentiometric titration to pH 4.0 (Standard Methods, 1975).

4.5.8 Gas Production

Each of the anaerobic filters was connected to a wet-test gas meter of the integrating type (Alexander Wright & Co. Ltd, England, 0.25 ℓ per revolution). Gas readings were taken daily for each unit and all measurements were corrected for volume at 30°C. The water in the meters was replaced and the meters rinsed approximately every month. After replacement of the water, the meters were reconnected to the filters and no readings taken for 48h to allow the water to become saturated with carbon dioxide.

4.5.9 Methane Composition of Gas

To measure the percentage of CH_4 in the digester gas, a sample was taken from the septum in gas line with a 1000 µℓ gas tight syringe (Scientific Glass Engineering Pty Ltd, Australia) and injected into a Varian Aerograph model 920 gas chromatograph fitted with a 1.52m x 6.5mm stainless steel column packed with 80/100 mesh Poropak S (Waters Associates Inc., U.S.A.) and a thermal conductivity detector. Methane peak heights were compared to a standard curve prepared by injecting 200, 400, 600, 800 and 1000 µℓ of 100 percent methane onto the column. The chromatograph operating conditions are listed in Table 4.3.

TABLE 4.3: Gas Chromatograph Conditions for Determination of Methane in Digester Gas

Carrier gas	dry nitrogen at 60mℓ/minute	
Reference gas	dry nitrogen at 12mℓ/minute	
Temperatures	injector	ambient
	column	80°C
	detector	120°C
Bridge current	90mA	

4.5.10 Volatile Acids

Gas-liquid chromatrography was used to measure volatile acid concentration. The method of Baufield *et al* (1978) was used. Chromatography conditions are listed in Table 4.4 The GLC was a Shimadzu GC5A with R101 Strip Chart recorder via a Varian CDS 111 digital integrator (Shimadzu Seisakusho Co. Ltd, Japan and Varian Aerograph, California, U.S.A. respectively). Mixed acid standard solutions were used to calibrate the instrument at each session and are described in Table 4.5.

Samples were first centrifuged to remove suspended material before proceeding with the analysis. The chromatograph

was fitted with a flame ionisation detector. Peak areas computed by the integrator were used to measure the volatile acid concentration of the samples.

TABLE 4.4: Gas Liquid Chromatographic Conditions for Volatile Acid Analysis

Carrier gas	dry nitrogen	at	80mℓ/minute
FID flows	hydrogen	at	50mℓ/minute
	air	at	900mℓ/minute
FID sensitivity			100 MΩ
Temperatures	injector		190°C
	column		160°C
	detector		190°C
Sample size			3 μℓ
Column size			2m x 5mm OD glass
Column packing			5% FFAP on 100/120 Chromosorb G.AW.DCMS

TABLE 4.5: Volatile Acid Working Standards

Component acid[1]	all values mg/ℓ Low standard	High standard
acetic	500	2000
propionic	500	2000
iso-butyric	100	1500
n-butyric	500	2000
iso-valeric	100	500
n-valeric	200	1000

[1]Analar grade BDH Chemicals, England in distilled water

CHAPTER FIVE

EXPERIMENTAL RESULTS

CHAPTER 5

EXPERIMENTAL RESULTS

This chapter gives the consolidated steady state experimental data obtained from the complete design. The anaerobic filter's effectiveness as a process to remove organic material and produce methane from yeast plant effluent is defined. In addition operational factors are calculated which characterise the process and the effluent.

5.1 Steady State Experimental Data

Data obtained from the twelve runs in the experimental design are presented in Table 5.1. These data were obtained using the methods detailed in the previous chapter. The first four columns in Table 5.1 refer to the combination of conditions prescribed in the experimental design; subsequent columns are mean values for the particular parameter over the period during which the filter was considered to be in a steady state with respect to those imposed conditions.

For the experimental work

- The BOD_5 of the feed was standardised. During the experimental period each batch of yeast plant effluent used was analysed for BOD_5 and diluted for use in the standardised basis. This parameter was used in the standardisation of the influent substrate concentration because the biochemical oxygen demand is of primary concern, along with suspended solids levels, to an authority assessing waste treatment costs.
- Gas production is the total gas production divided by initial void volume of the filters, so as to standardise this parameter with respect to anaerobic filter size.
- Incomplete BOD_5 data were due to large disagreement between sample duplicates for runs 7 and 8. The four duplicated samples for run 7 were 2163 and 1047 mg/ℓ,

2092 and 1546 mg/ℓ, 1124 and 2120 mg/ℓ, 2023 and 1876 mg/ℓ respectively taken over the steady state period. For run 8, they were 1040 and 3970 mg/ℓ, 1539 and 1844 mg/ℓ, 1457 and 1916 mg/ℓ, 1779 and 1130 mg/ℓ. For run 5, the sample was lost in the laboratory. No satisfactory answer was found for the disparity in the duplicates for runs 7 and 8. The obvious one of leakage from the manometric BOD bottles was not the cause as all seals were checked prior to analysis. Agreement between duplicates of at least 5 percent was found for the other samples.

- Incomplete sulphide data was caused by the non-availability of a sulphide specific ion probe for runs 1, 2, 4 and 5. Samples taken for other analyses during these runs did smell of sulphide, as did all other samples, so sulphides were present even if concentrations are unknown.

- The suspended solids data for runs 1, 2, 4 and 5 are residue on evaporation values not suspended solids.

In order to ascertain the degree of association between the input and response variables in Table 5.1, correlation coefficients were computed (Table 5.2). The calculations used to generate Table 5.2 took into account the missing data points in Table 5.1. Also the significance levels are based on n-2 degrees of freedom where n is the number of pairs of data used in calculating the correlation coefficient. The correlations that were statistically significant with confidence levels of 95 and 99 percent are marked in the table. The table provides information as to possible relationships between variables. It also provides statistics for data presented graphically later in this chapter.

In addition to the data from the runs in the experimental design used in this study, data are presented in Table 5.3 for the two trials already completed in the first design but dropped when the programme was shortened (see 4.2.2).

In the next sections, the effects of each of the four input variables, and the hydraulic loading rate, on various

TABLE 5.1: Steady State Data from the Experimental Design

	RUN NUMBER	1	2	3	4	5	6	7	8	9	10	11	12
design	INFLUENT BOD (mg/ℓ)	1000	1000	1000	1000	5000	5000	5000	5000	9000	9000	9000	9000
	HYDRAULIC RETENTION TIME (d)	1	1	3	5	1	3	3	5	1	3	5	5
	TEMPERATURE (^{o}C)	30	30	35	35	35	30	35	30	35	30	30	35
	FILTER UNIT	1	2	2	1	2	1	2	1	1	1	2	2
	organic loading rate kg BOD_5/m^3d	1.0	1.0	0.33	0.20	5.0	1.67	1.67	1.0	9.0	3.0	1.8	1.8
influent	COD mg/ℓ	5486	5486	5893	5773	27806	24309	24929	24242	47175	46462	46462	43805
	total nitrogen mg/ℓ	110	110	106	135	573	550	526	532	930	1126	1126	1090
	total sulphate	386	386	334	436	2180	1955	1864	1812	3410	3100	3100	2746
	pH	4.80	4.80	4.95	5.00	5.95	5.95	5.80	5.80	5.65	5.70	5.55	5.50
	organic loading rate kg COD/m^3d	5.49	5.49	1.96	1.15	27.8	8.70	8.31	4.85	47.2	15.49	9.29	8.76
	gas production ℓ/ℓd	0.845	0.599	0.409	0.773	1.770	1.296	2.010	1.350	3.110	2.153	1.640	1.784
	% CH_4 in gas	83.5	86.7	82.05	80.0	75.9	66.3	66.5	67.9	49.9	56.4	67.4	73.4
	CH_4 production ℓ/ℓd	0.706	0.519	0.335	0.138	1.343	0.859	1.337	0.917	1.552	1.214	1.105	1.309
effluent	COD mg/ℓ	2153	1888	1931	2825	17378	13642	9879	8978	31090	30862	27677	25508
	BOD mg/ℓ	628	539	567	348	-	2037	-	-	4455	2823	4828	4435
	pH	685	6.75	6.95	7.00	6.90	6.85	7.25	7.25	6.90	6.95	7.05	7.05
	volatile acids mg/ℓ[1]	714	672	342	390	3737	1811	2096	864	4846	2843	2020	2420
	ammonia mM	2.37	2.35	9.15	-	18.1	-	9.7	10.5	6.6	5.7	8.9	-
	sulphide mg/ℓ	-	-	54	-	-	104	169	131	186	253	150	99.5
	alkalinity mg/ℓ[2]	1445	1430	1870	1450	3896	3527	4383	4475	6860	6116	6310	6189

con'd over next page

suspended solids mg/ℓ	2963[5]	1735[5]	320	2350[5]	3342[5]	190	850	260	740	590	795	816
% COD removed	60.8	65.6	67.2	51.1	37.5	43.9	60.3	63.0	34.1	33.6	40.4	43.0
% BOD removed	37.2	46.1	43.3	65.2	-	59.3	-	-	50.5	68.6	46.4	50.7
COD removal rate[3]	3.336	3.599	1.320	0.590	10.43	3.557	5.019	3.054	16.08	5.204	3.754	3.767
FTCM[4]	0.541	0.369	0.651	0.600	0.531	0.618	0.682	0.772	0.246	0.597	0.756	0.687

Notes:
1 total volatile acids mg/ℓ as acetic
2 total alkalinity mg/ℓ as Ca CO_3
3 kg/m^3d
4 fraction of the theoretical maximum conversion of COD to methane
5 total solids value

TABLE 5.2: Correlation Coefficients for Data in Table 5.1 (x 1000)

___ = correlation significant P < 0.01

	BOD (IN)	hydraulic retention	temperature	AF unit	COD (IN)	total nitrogen	total sulphate	pH	organic loading (COD)	organic loading (BOD)	COD	BOD	% COD removed	COD removal rate	% BOD removed	gas production	% methane in gas	methane production	FTCM	pH	volatile acids	ammonia	sulphide	alkalinity
hydraulic retention	250																							
temperature	000	000																						
AF unit	000	000	333																					
COD (IN)	998	212	015	005																				
total nitrogen	991	302	-041	031	988																			
total sulphate	986	158	017	-036	990	964																		
pH	686	156	049	-069	681	641	735																	
organic loading (COD)	541	-502	307	-137	580	462	637	437																
organic loading (BOD)	563	-483	300	-150	699	483	656	450	999															
COD (OUT)	969	114	025	-039	981	966	969	606	645	662														
BOD (OUT)	995	304	078	149	953	939	943	723	591	605	930													
% COD removed	-782	-004	-096	188	-806	-787	-822	-579	-667	-673	881	-308												
COD removal rate	501	558	301	-081	538	414	602	445	989	987	587	567	-589											
% BOD removed	293	395	046	-482	291	315	315	514	064	074	327	070	-572	-023										
gas production	642	-313	260	-218	662	550	702	428	912	927	684	657	-570	903	044									
% methane in gas	-817	-070	-004	376	-822	-760	-860	-713	-653	-679	-805	-731	675	-614	-454	-801								
methane production	686	320	272	-127	704	596	745	493	917	930	713	718	-587	922	-005	987	-782							
FTCM	079	836	-234	052	034	135	-019	092	-720	-702	-093	094	276	-734	104	-501	057	485						
pH	279	662	199	085	251	262	247	417	-207	-192	085	530	150	-177	234	005	-277	043	674					
volatile acids	951	126	119	022	952	916	946	589	674	696	930	975	-737	649	111	787	-785	828	-091	212				
ammonia	221	197	541	368	237	202	297	708	240	221	177	514	-280	253	141	020	-108	103	035	390	163			
sulphide	643	-305	-283	-441	668	617	706	492	497	500	660	417	-600	489	690	493	-839	490	-332	032	479	-704		
alkalinity	988	246	056	009	987	966	979	691	574	597	945	963	-721	542	244	697	-844	738	077	360	960	250	665	
suspended solids	640	-007	436	490	648	626	595	-003	358	361	584	891	-392	389	-174	426	-287	504	-170	234	690	-310	415	691

TABLE 5.3: Steady State Data from Runs not Included in the Experimental Design

Run	'A'	'B'
Influent substrate (mg BOD_5/ℓ) concentration	1000	5000
Hydraulic retention time (d)	5	1
Temperature (^oC)	30	35
Filter unit	2	1
Organic loading rate (kg BOD_5/m^3d)	0.20	5.0
Influent COD (mg/ℓ)	5773	28245
Total nitrogen (mg/ℓ)	135	584
Total sulphate (mg/ℓ)	436	2075
pH	5.0	5.75
Organic loading rate (kg COD/m^3d)	1.15	28.2
Gas production (ℓ/ℓd)	.198	1.069
CH_4 in gas (%)	73.0	71.25
CH_4 production (ℓ/ℓd)	0.149	0.762
BOD (mg/ℓ)	451	1580
COD (mg/ℓ)	3005	10145
Ammonia (nM)	- *	19.1
Sulphide (mg/ℓ)	- *	- *
Alkalinity (mg/ℓ as $CaCO_3$)	1162	3393
Suspended solids (mg/ℓ)	205	187
Volatile acids (mg/ℓ as acetic)	500	1440
% COD removed	47.9	64.1
% BOD removed	54.9	32.1
COD removal rate (kg COD/m^3d)	0.551	18.07
FTCM	0.714	0.139

* probes unavailable.

system responses are assessed. The trends in the data are shown graphically. In the graphs, the experimental data are plotted together with predicted response curves. These latter curves are the relevant reponse equations calculated from the data in Table 5.1. Thus an indication of the ratio of actual to predicted behaviour may be made visually.

The effects of two factors, influent substrate concentration and hydraulic retention time, only are shown; and also the combination of these two, the organic loading rate. This is because the effects of the temperature and filter size were found to be non-significant statistically in most cases.

In the following figures, important system responses are illustrated with respect to the organic loading rate composite parameter, followed by the resolution of this to its influent substrate concentration and hydraulic retention time components.

5.2 The Effect of the Experimental Factors on the Removal of COD and BOD_5

5.2.1 Removal of Soluble COD

The soluble COD removal from the system is represented in terms of effluent COD concentrations, percentage COD removal and removal rate.

Effluent COD

The effect of organic loading rate on effluent COD concentration is shown in Figures 5.1 and 5.1a. There is a general trend towards increased effluent COD at higher organic loading rate; however, the runs at constant influent substrate concentration and constant retention time indicated the large effect of the former and the relative independence of the latter.

The three runs carried out at an equal organic loading rate of 1 kg BOD/m^3d, but different concentration retention

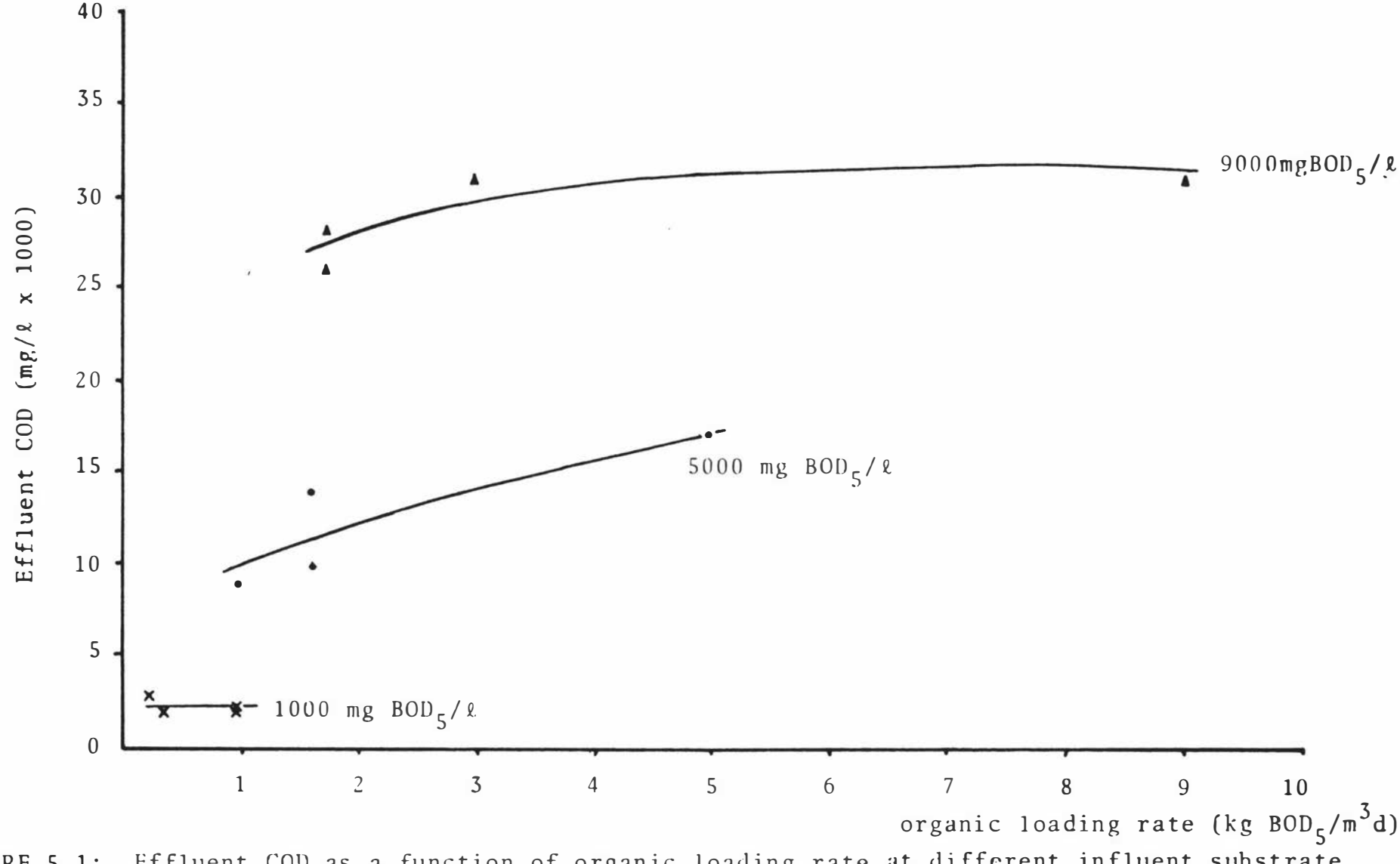


FIGURE 5.1: Effluent COD as a function of organic loading rate at different influent substrate concentrations

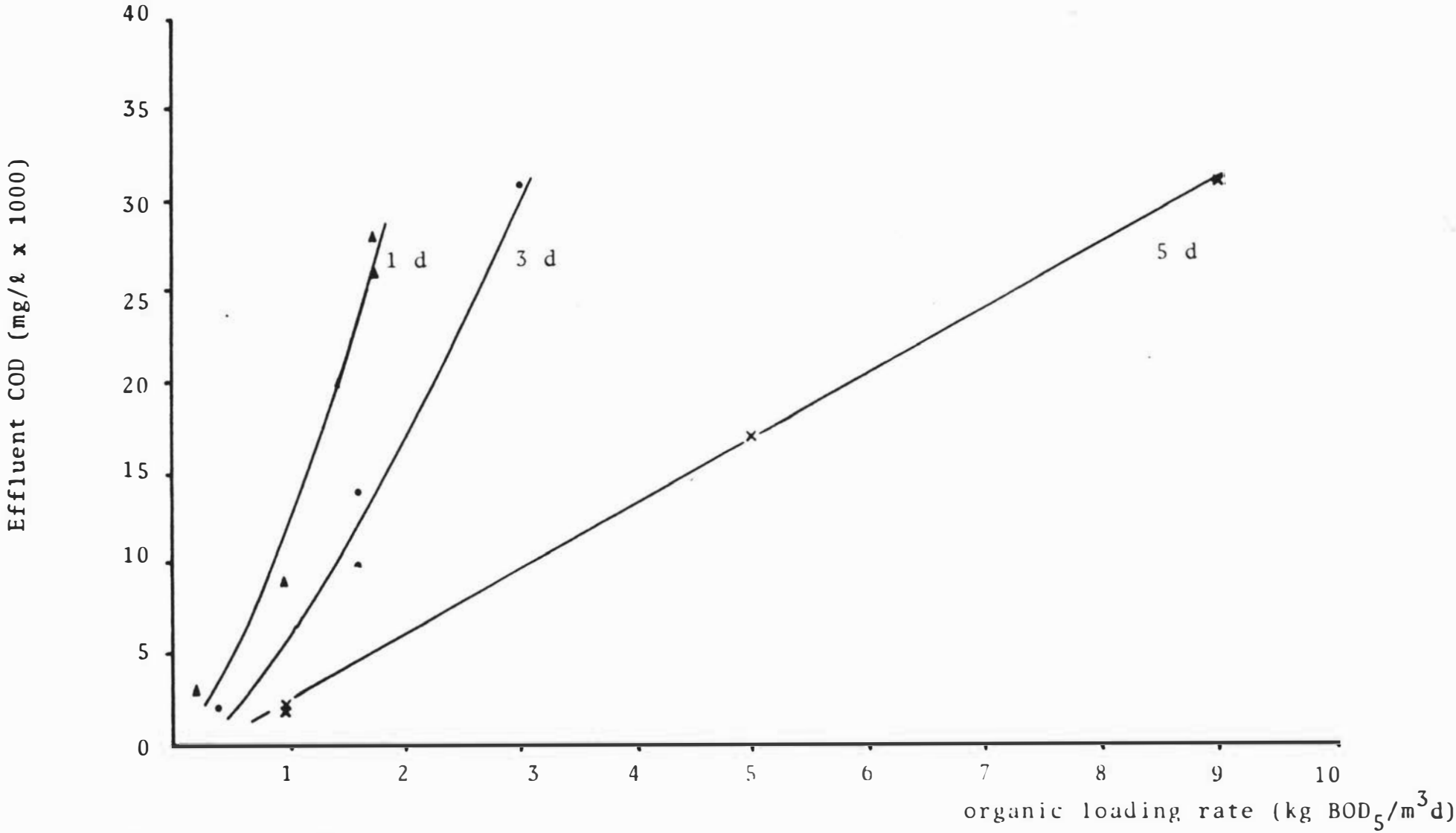


FIGURE 5.1a: Effluent COD as a function of organic loading rate at different HRT

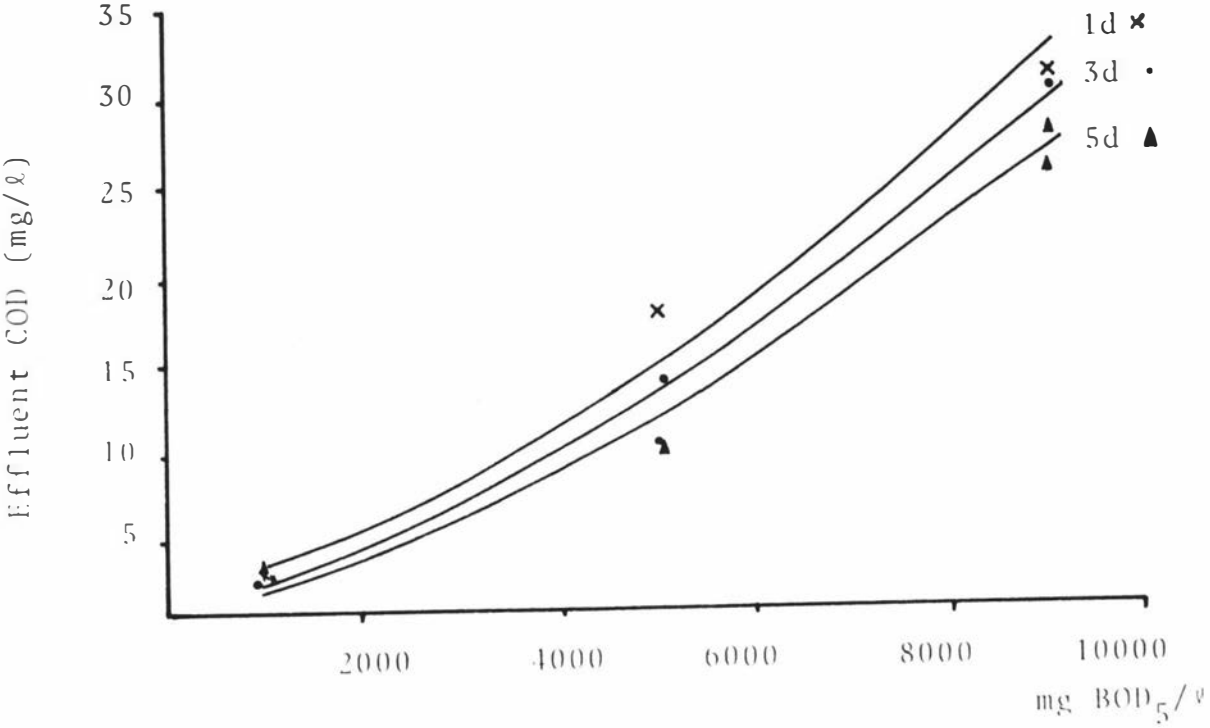


FIGURE 5.2: Effect of influent substrate concentration on effluent COD at different HRT

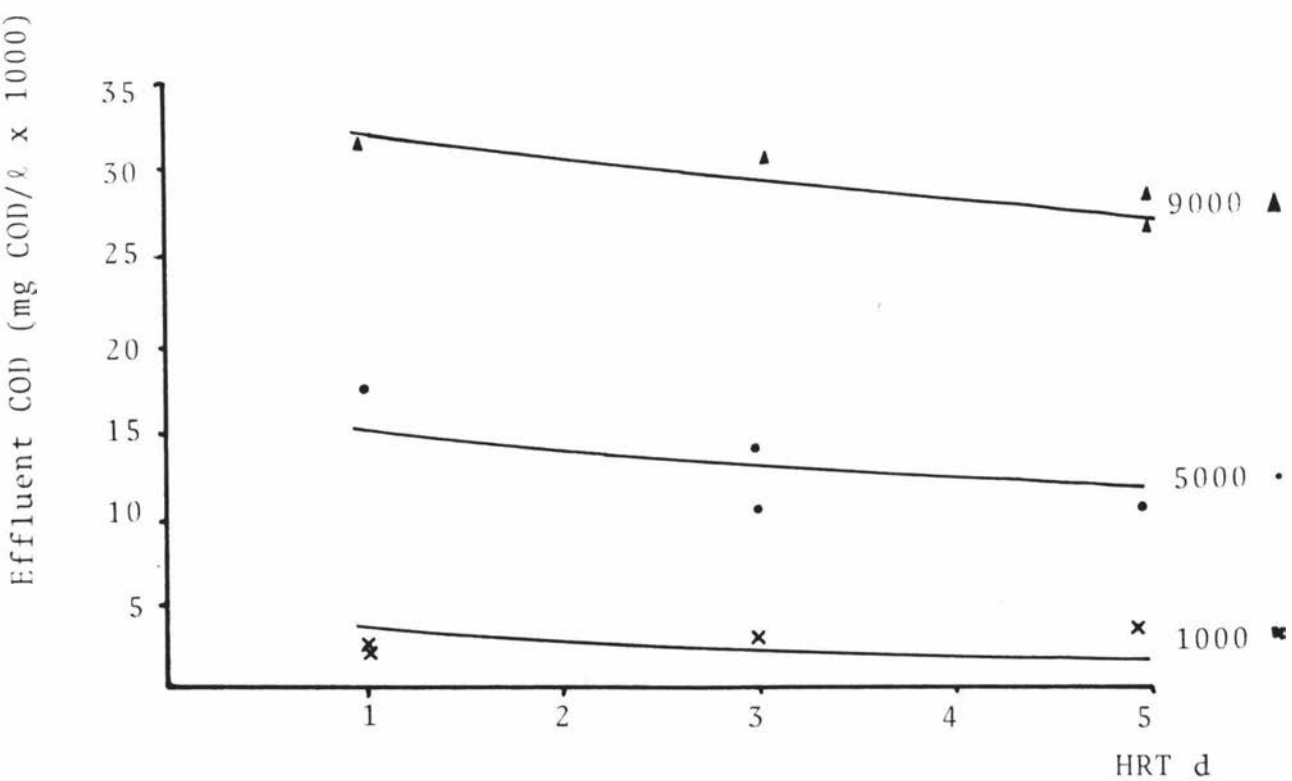


FIGURE 5.3: Effect of hydraulic retention time on effluent COD at different influent substrate concentrations

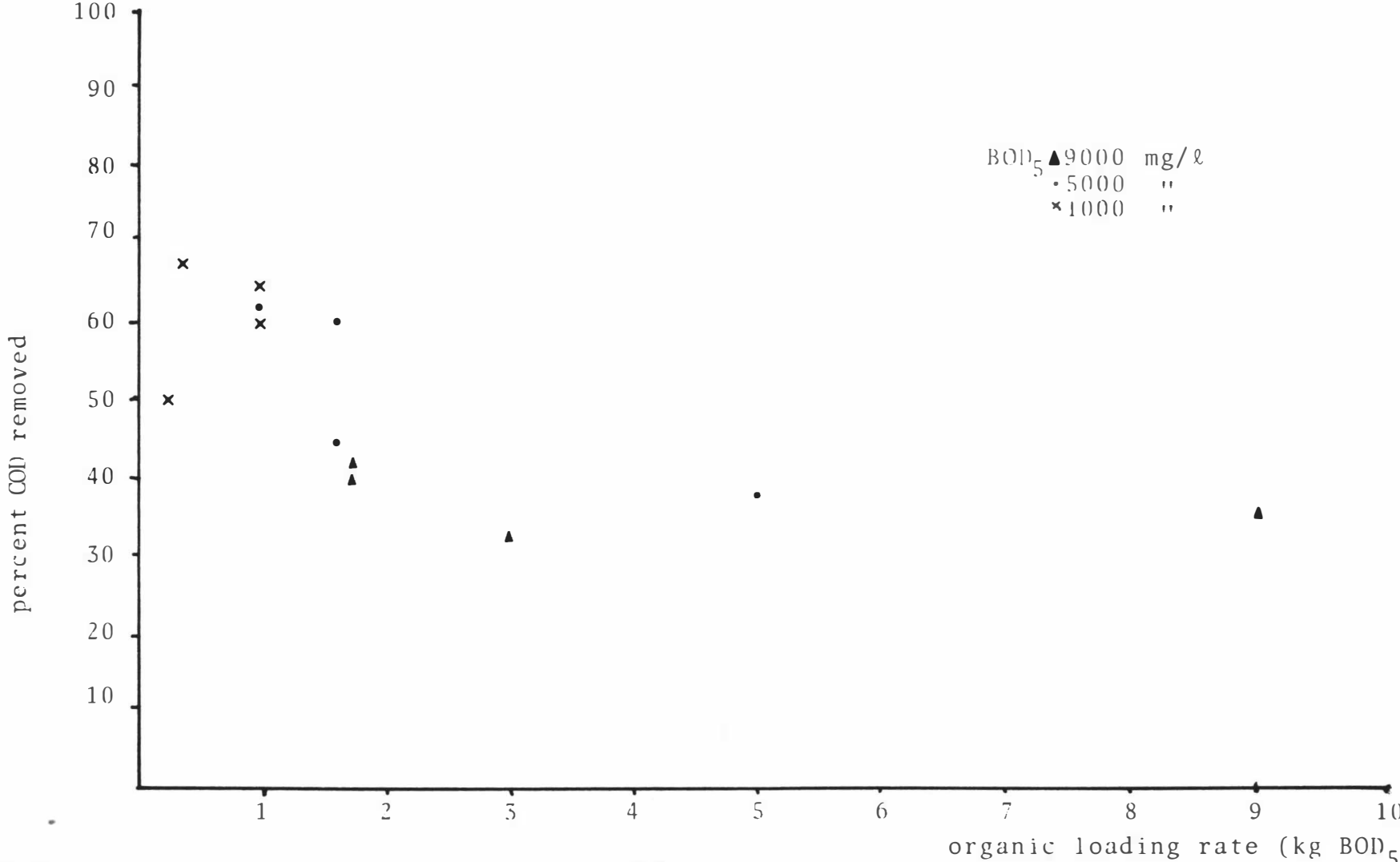


FIGURE 5.4: Percent COD removal as a function of organic loading rate at different influent substrate concentrations

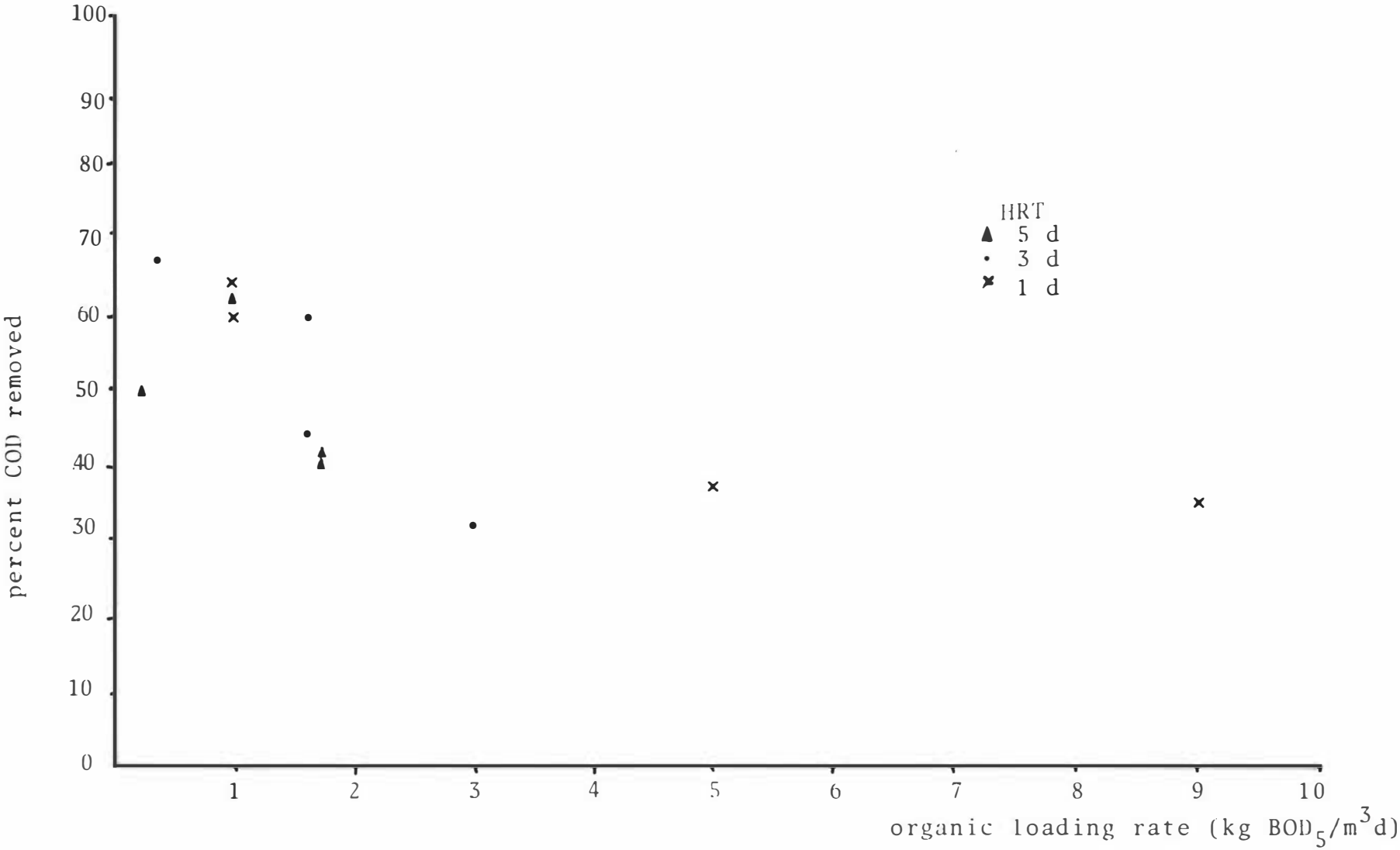


FIGURE 5.4a: Percent COD removal as a function of organic loading at different HRT

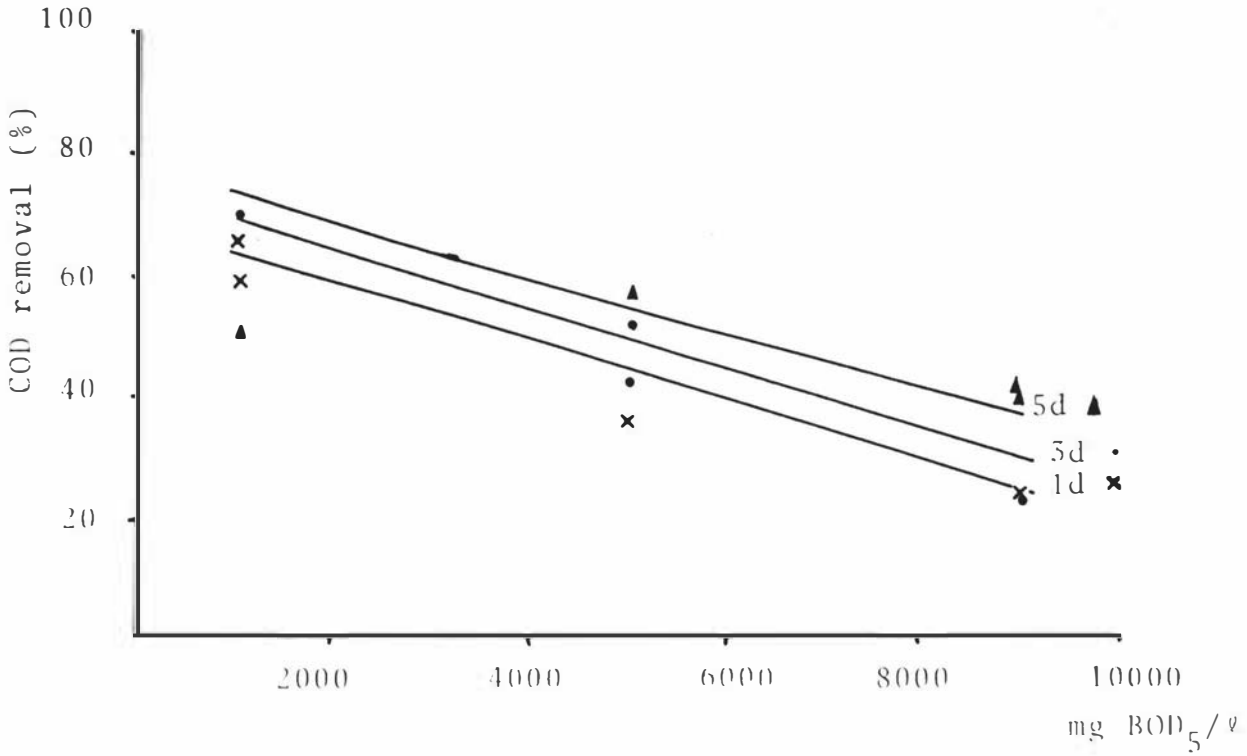


FIGURE 5.5: Effect of influent substrate concentration on percent COD removed at different HRT

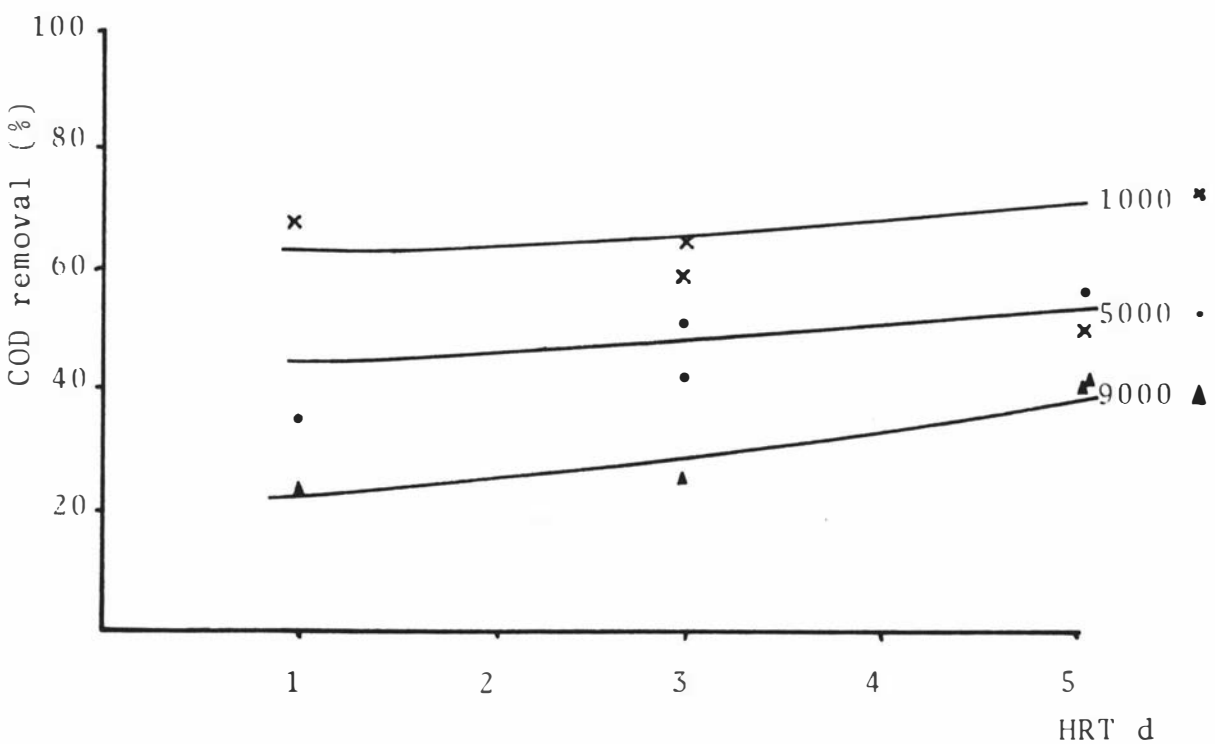


FIGURE 5.6: Effect of HRT on percent COD removed at different influent substrate concentration

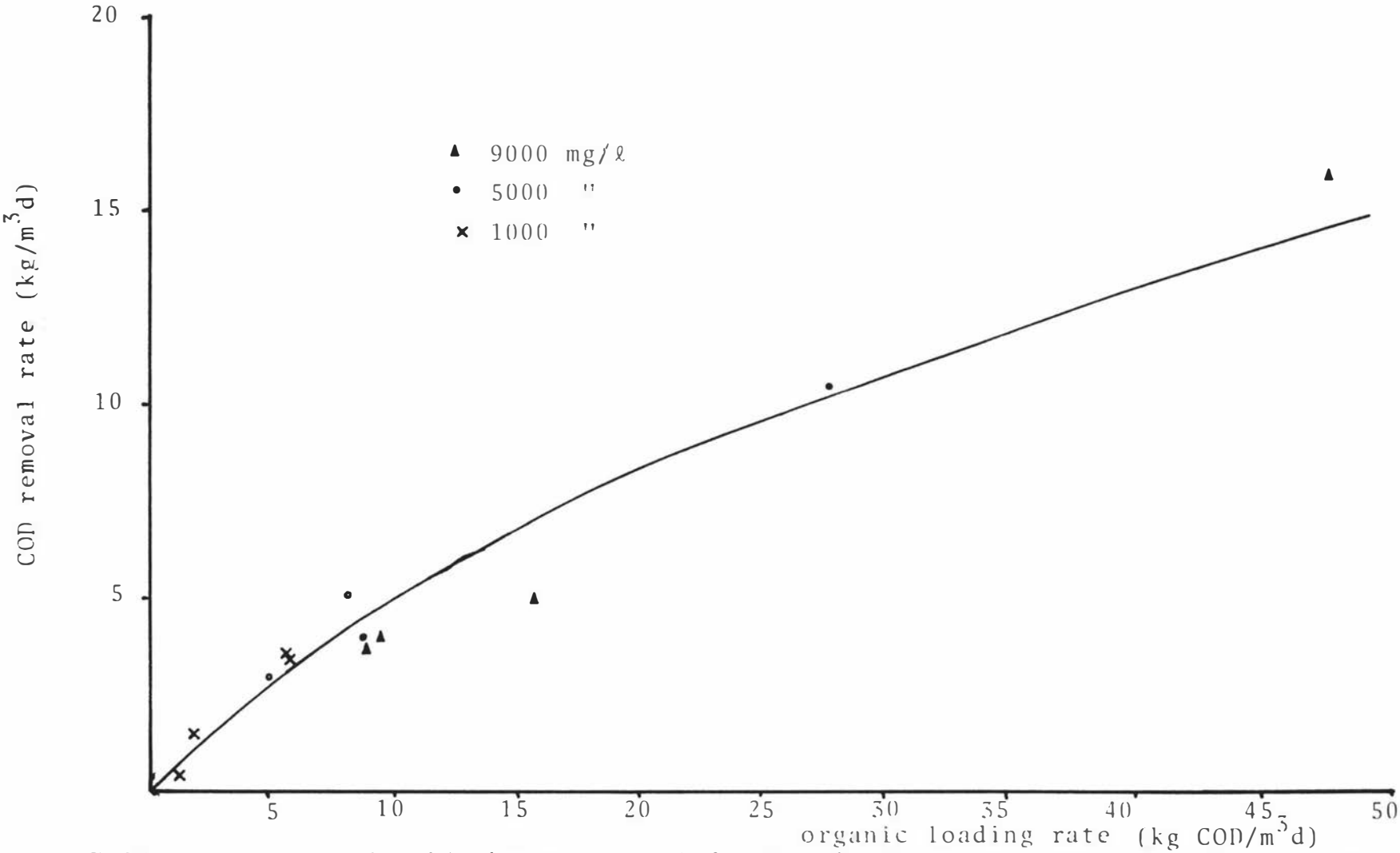


FIGURE 5.7: Relationship between applied COD and COD removal rate at different influent substrate concentrations

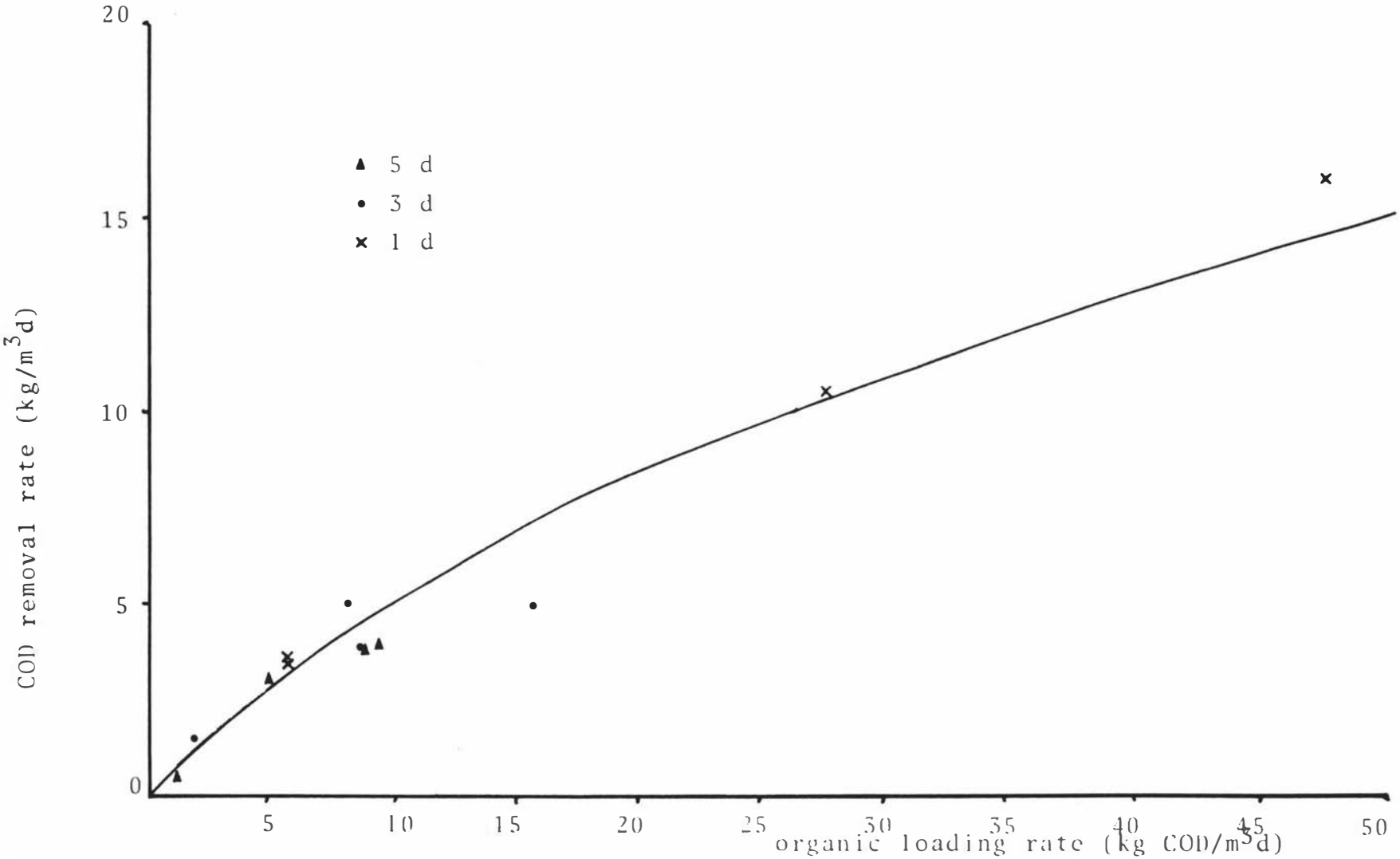


FIGURE 5.7a: Relationship between applied COD and COD removal rate at different hydraulic retention times.

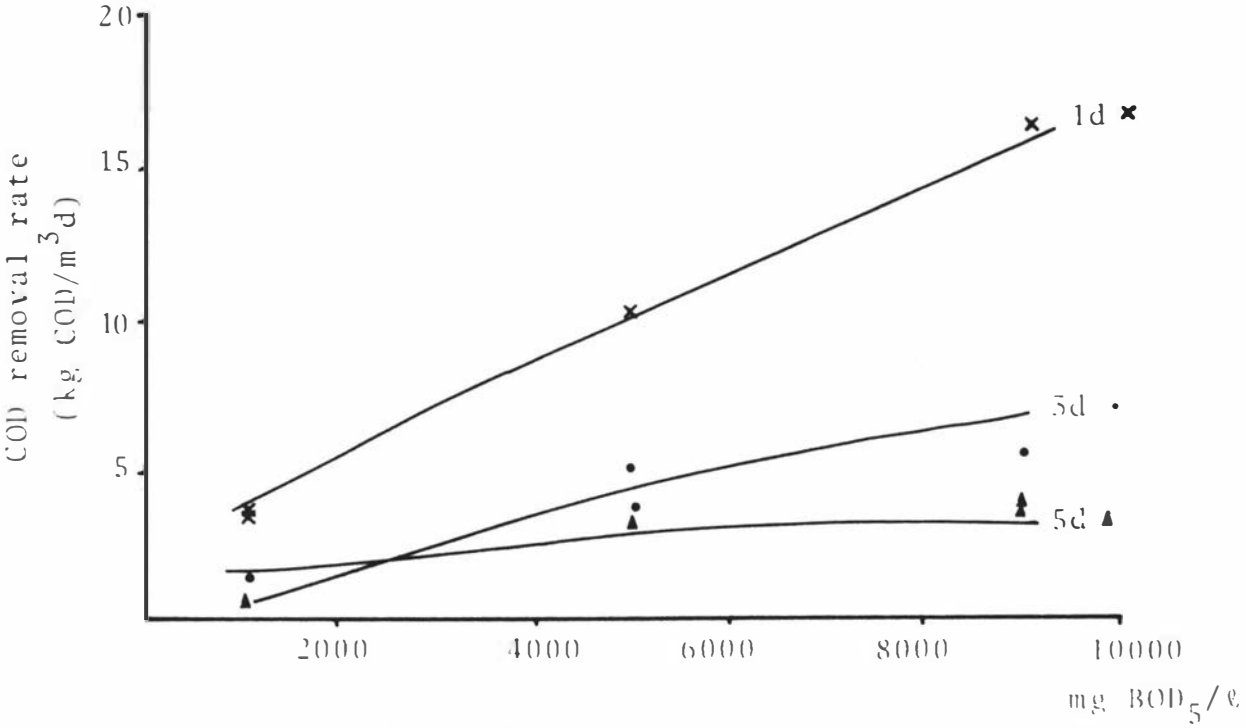


FIGURE 5.8: Effect of influent substrate concentration on COD removal rate at three HRT

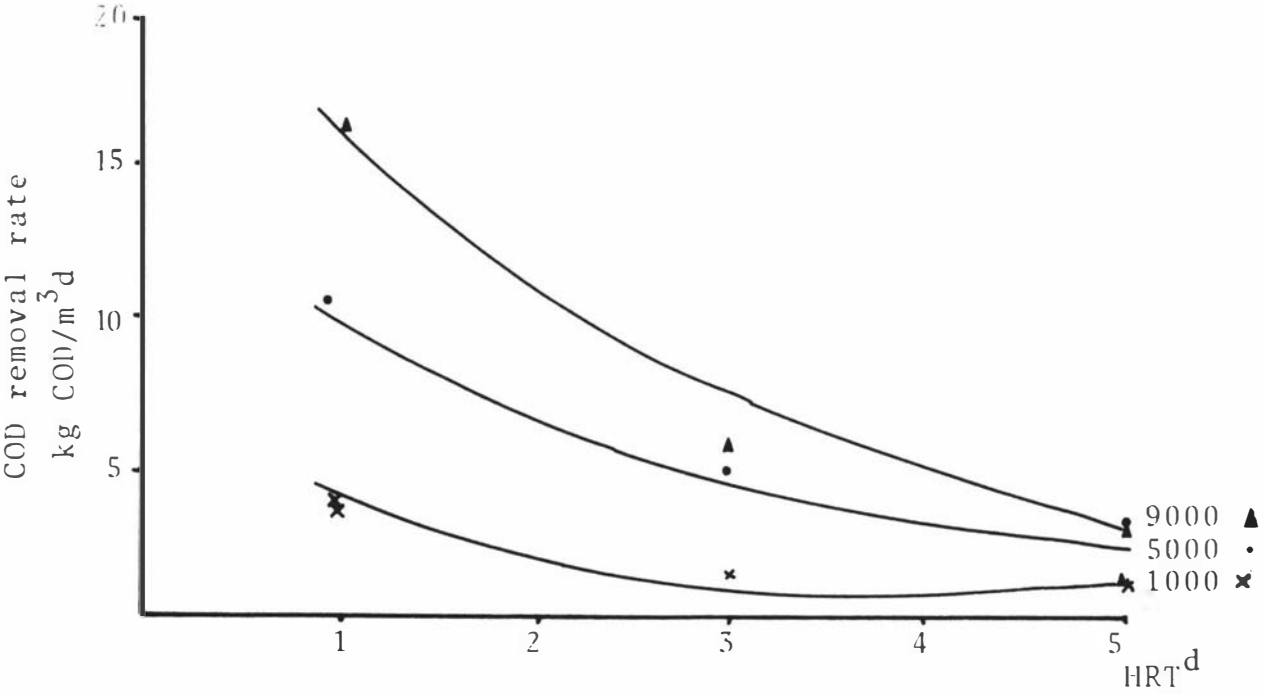


FIGURE 5.9: Effect of hydraulic retention time on COD removal rate at three influent substrate concentrations

times, indicated that for this response parameter, the equal organic loading rates do not give rise to equivalent effluent COD.

The individual effects of influent substrate concentration and hydraulic retention time on effluent COD are indicated in Figures 5.2 and 5.3 respectively. There is little advantage in terms of effluent quality in extending the hydraulic retention time beyond one day at each level of influent substrate concentration. The regression model (Equation 5) shows good fit to the observed data in terms of both parameters although there is considerable spread in the midpoint (5000 mg BOD_5/ℓ) of the influent substrate concentration.

Percentage Removal

The effects of the three parameters on percentage removal of COD are presented in Figures 5.4 to 5.6. There was a general trend towards lower percentage COD removal at higher loading rates (Figure 5.4). There was considerable spread in the data however, especially at the 1000 and 5000 mg BOD_5/ℓ levels (Figure 5.4a). At 9000 mg BOD_5/ℓ influent substrate concentration, higher loading rates were accompanied by reduced percentage COD removals, with an average of 41.7 percent removal at 1.8 kg $BOD_5/\ m^3d$ reducing to 34.1 percent at 9 kg BOD_5/m^3d.

Hydraulic retention time only exerted an effect on percentage COD removal at loading rates below about 3 kg BOD_5/m^3d. For this response parameter, there was good agreement between the runs carried out at equal loading rate of 1 kg BOD_5/m^3d, with an average 63.2 percent of removal at 1000 mg BOD_5/ℓ and 1 d; and 63.0 percent at 5000 mg BOD_5/ℓ and 5 d.

The individual effects of influent substrate concentration and HRT are presented in Figures 5.5 and 5.6 respectively. The relative independence of percent COD removal of retention time is well illustrated in Figure 5.6 with only small increases in removal with retention times longer than 1 day.

The regression lines give reasonable fit to the experimental data except for the run at 1000 mg BOD_5/1 and 1 d HRT. This was most likely a non-typical run in that it was only one point in twelve that was outside the trend of the data.

Rate of COD Removal

In Figures 5.7 and 5.7a, the rate of organic material removed is related to its application rate. An apparently linear relationship at first glance is due to the effect of scale only. Rate of removal is expressed as $(S_o - S)/t$ and organic loading rate as S_o/t, hence the slope on the Figure 5.7 is $(S_o - S)/S_o$ if linear. This term is the definition of the fraction of COD removed which is clearly not constant at constant influent substrate concentration or HRT (Figures 5.5 and 5.6 respectively).

When the effects of substrate concentration and hydraulic retention time are considered separately, as in Figures 5.8 and 5.9 respectively, the effect of a reduction in retention time is apparent. At a 5d HRT, a nine fold increase in influent substrate concentration gave rise to a 6.3 fold increase in removal rate and at 1 d HRT, the increase was 3.37 kg COD/m^3d to 16.08 kg COD/m^3d, a 4.7 fold increase.

It will be seen later that at the shorter retention time there is less conversion of COD to methane. The equal organic loading rate runs agreed well with each other in respect of rate of removal of COD, with 3.37 kg COD removed/m^3 d at 1000 mg BOD_5/ℓ and 1d HRT and a slightly higher removal rate of 3.56 kg COD/m^3d at the 5000 mg BOD_5/ℓ and 5 d HRT combination.

5.2.2 Temperature Effects on Substrate Removal

No effect due to temperature could be assigned using the data in the preceding section. However, by maintaining the assumption that the effects due to the individual filter unit used are negligible and manipulating the

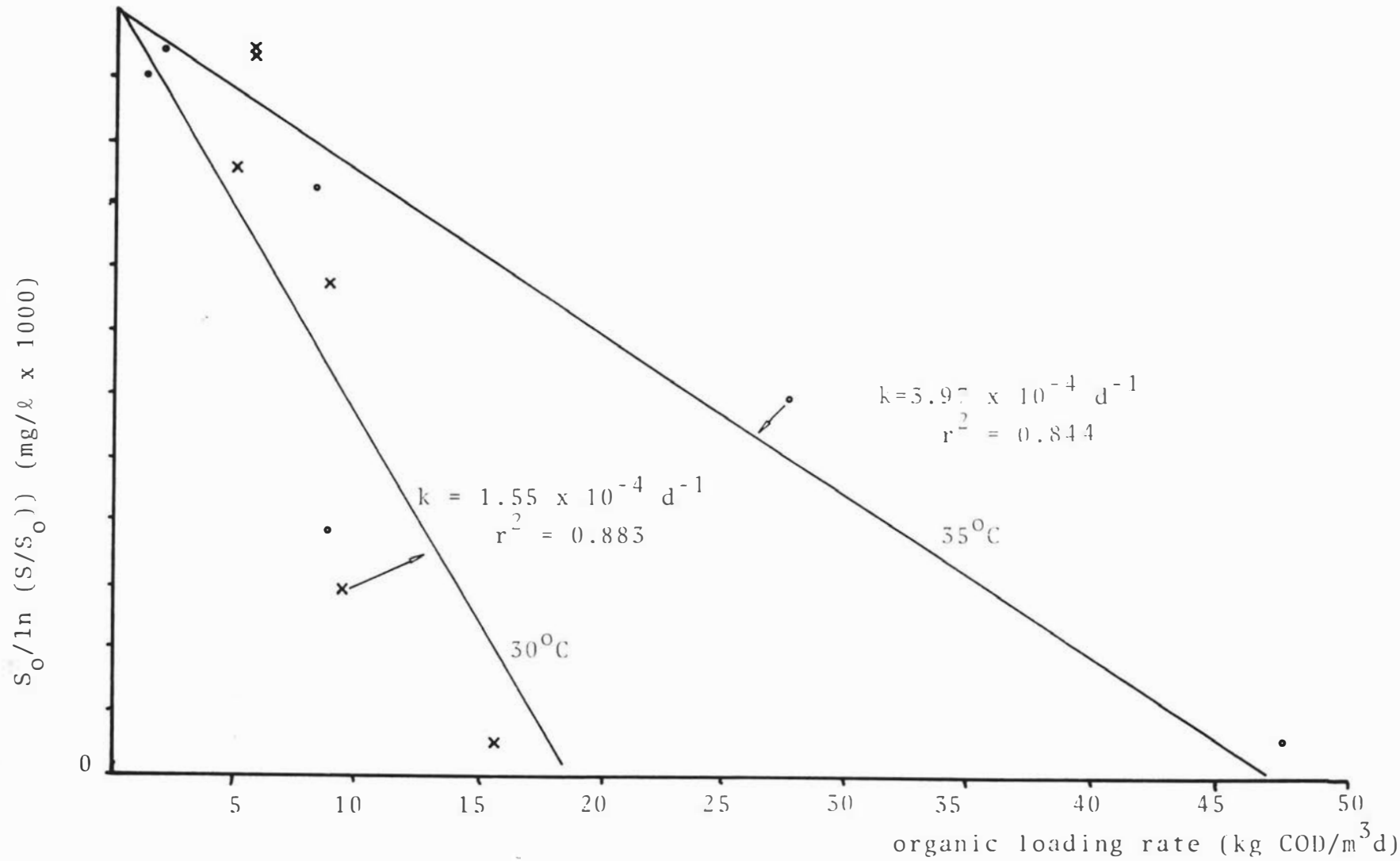


FIGURE 5.10: The effect of temperature on COD removal

substrate data into a form of equation 2.13 a temperature effect on substrate removal kinetics becomes apparent. Figure 5.10 shows that temperature may indeed have had expected positive effect on removal rate of COD. The coefficients of determination (r^2) for both regression lines are reasonably high and the magnitude of the k values indicate an increase in removal rate of about 2.5 fold for the $5^{o}C$ temperature rise and E_a of 140 kJ.

The scatter of data points about the regression lines is not random. For the $35^{o}C$ plot the regression line overestimates the actual data at low organic loading rates and underestimates it at higher, while this trend is consistently reversed for the $30^{o}C$ line. This is possibly due to the first order approximation used being an inappropriate model to adequately explain the observed variation.

5.2.3 Removal of BOD

The effect of influent substrate concentration, hydraulic retention time and organic loading rate on removal of BOD from the yeast plant effluent are illustrated in Figures 5.11 to 5.17.

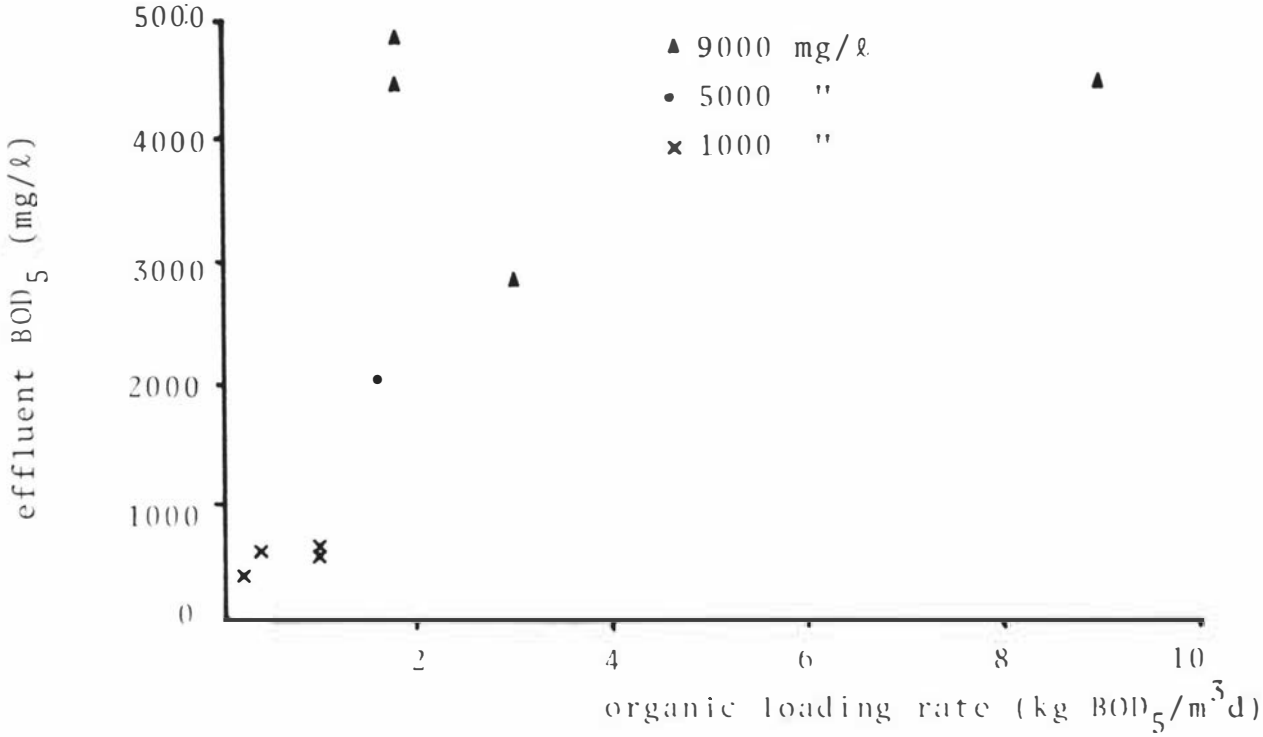


FIGURE 5.11: Effluent BOD_5 as a function of organic loading rate at different influent substrate concentrations

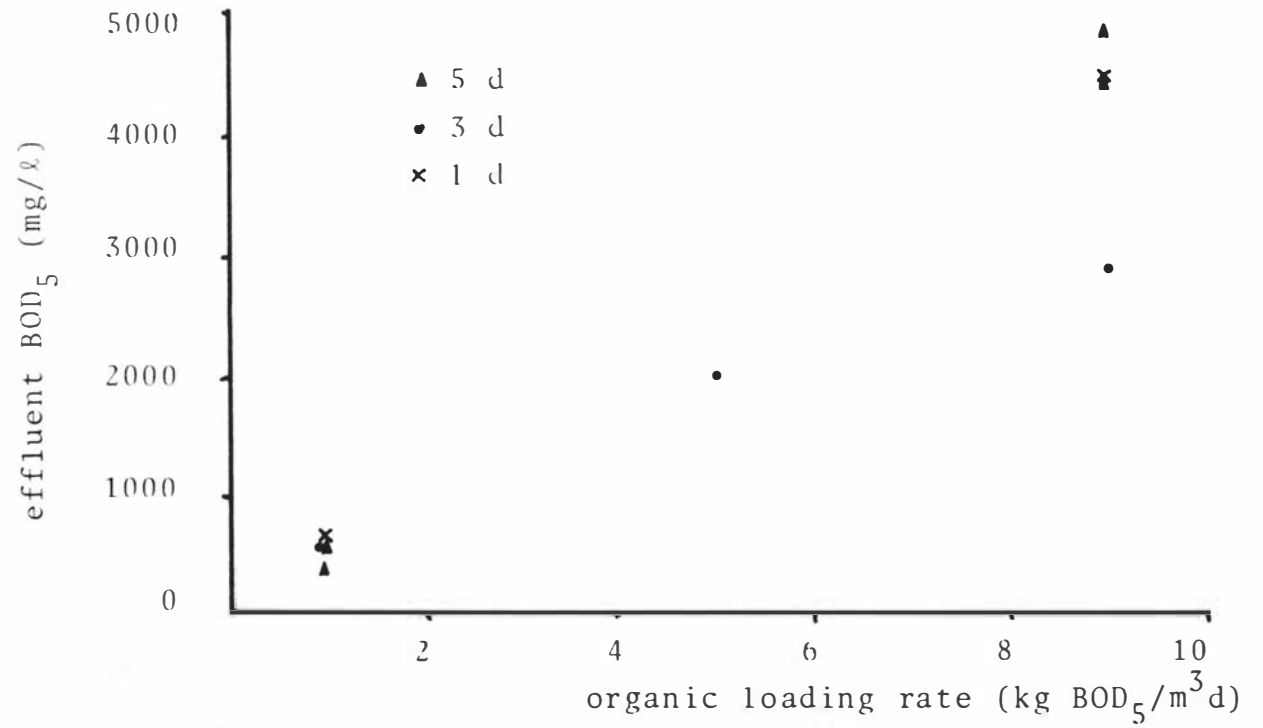


FIGURE 5.12: Effect of influent substrate concentration on effluent BOD_5 at different hydraulic retention times

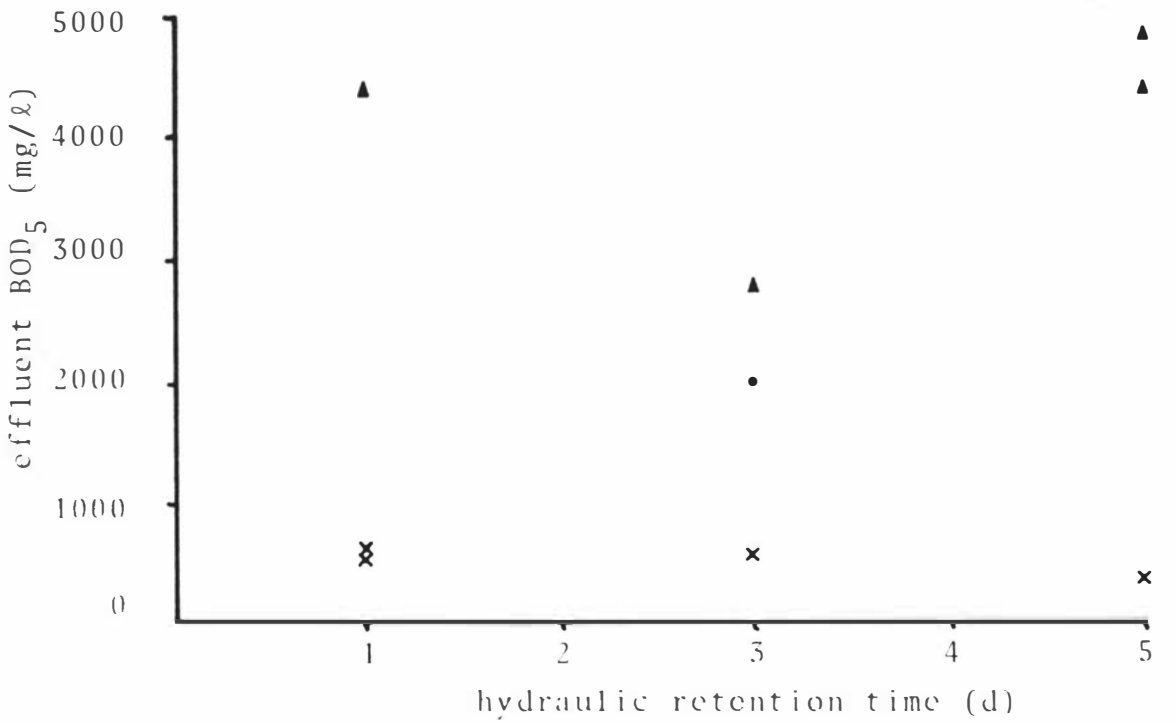


FIGURE 5.13: Effect of HRT on effluent BOD_5 at different influent substrate concentrations

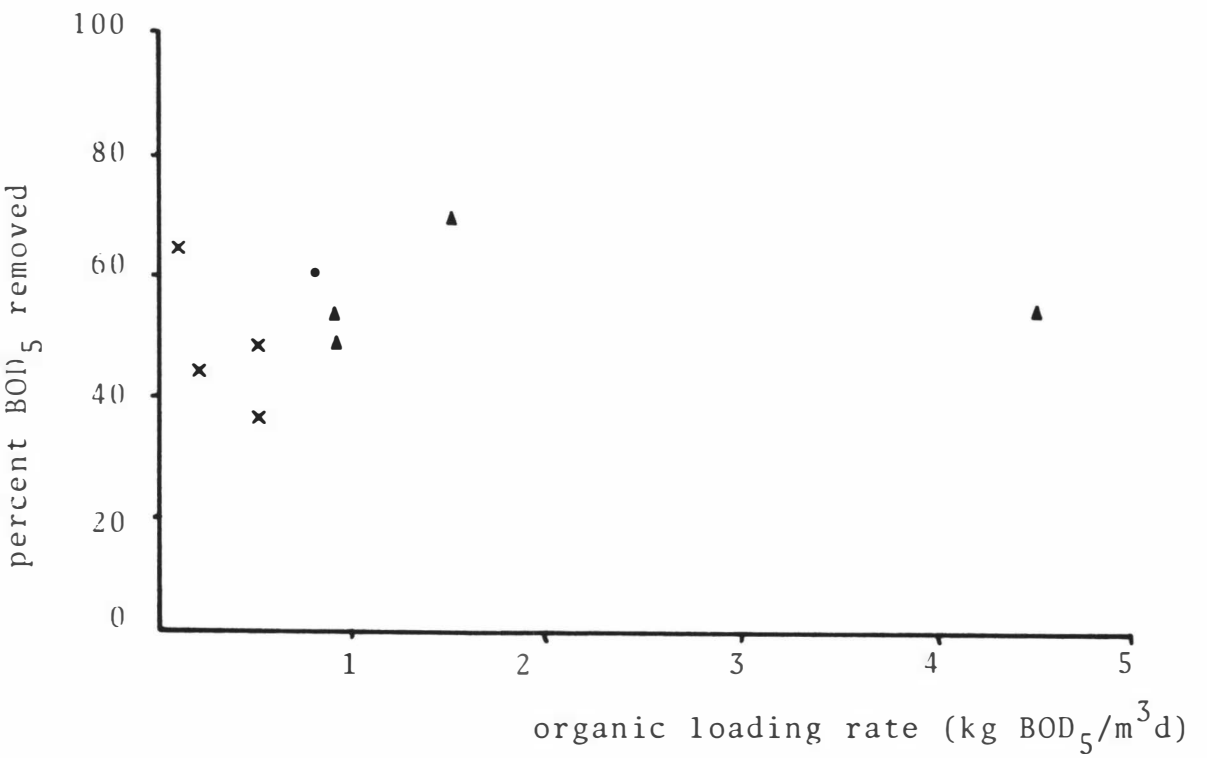


FIGURE 5.14: Percent BOD_5 removal as a function of organic loading rate at different influent substrate concentrations

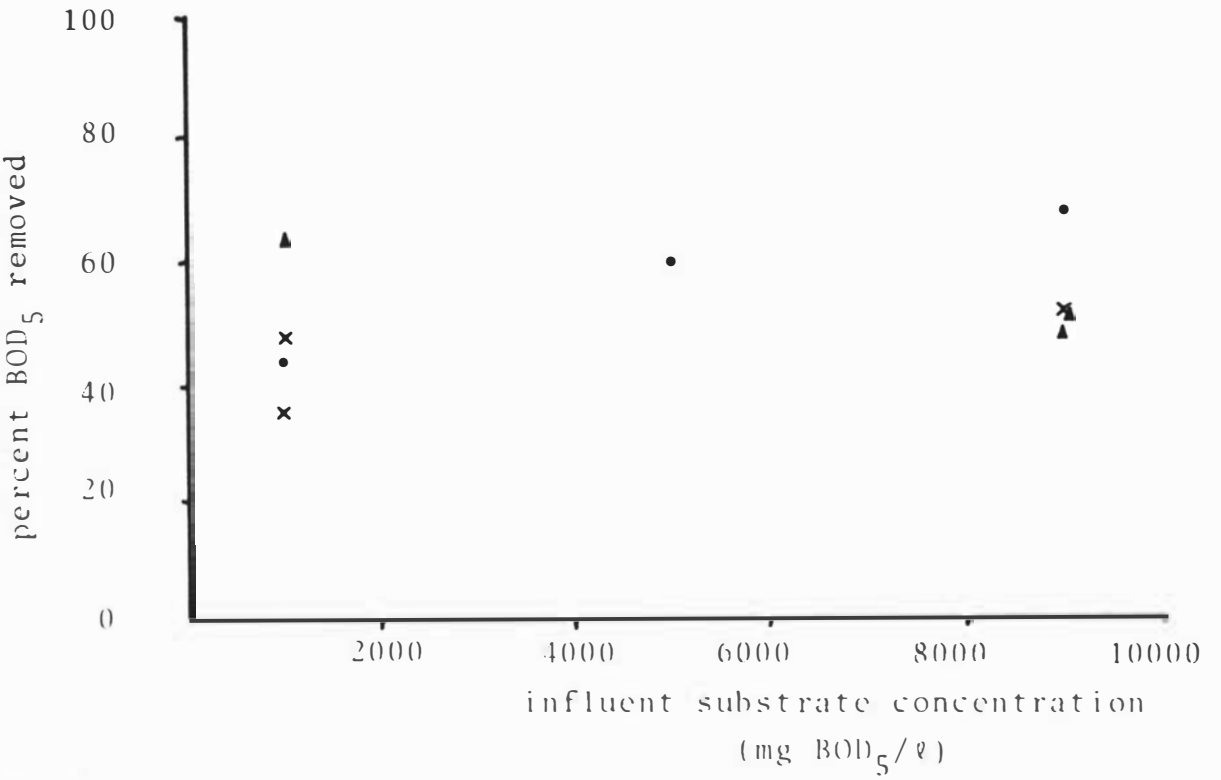


FIGURE 5.15: Effect of influent substrate concentration on percent BOD_5 removal at different hydraulic retention times

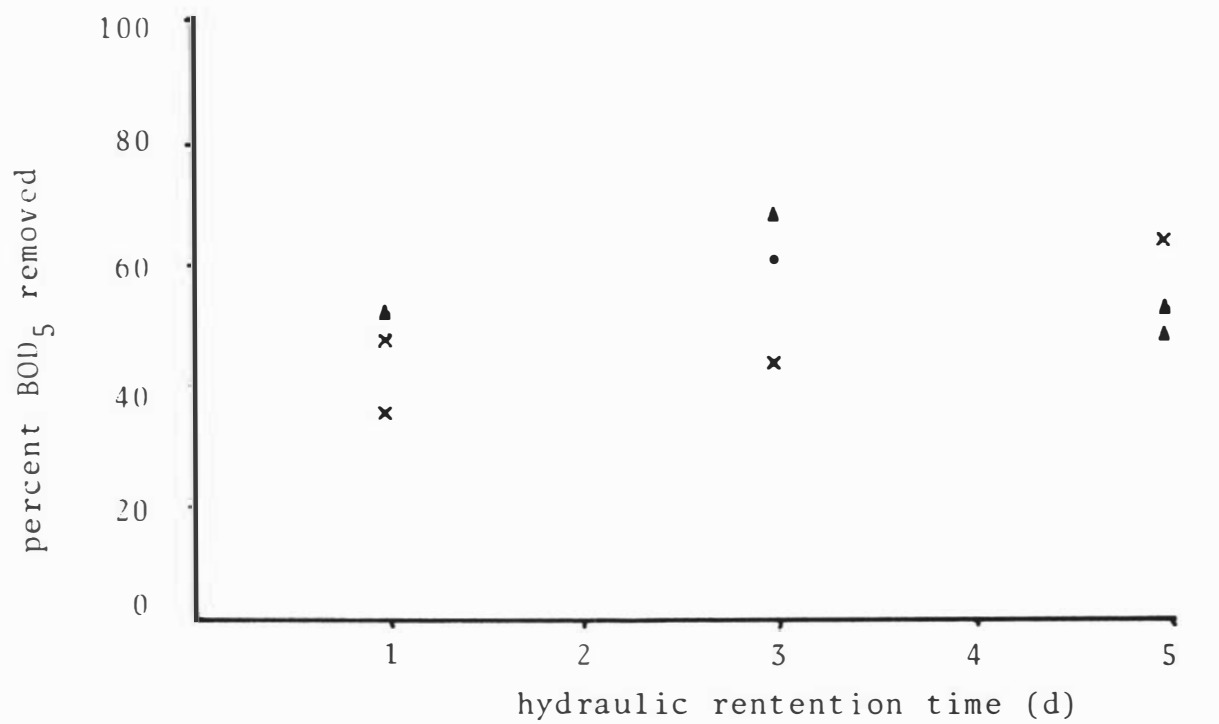


FIGURE 5.16: Effect of hydraulic retention times on percent BOD_5 removed at different influent substrate concentrations

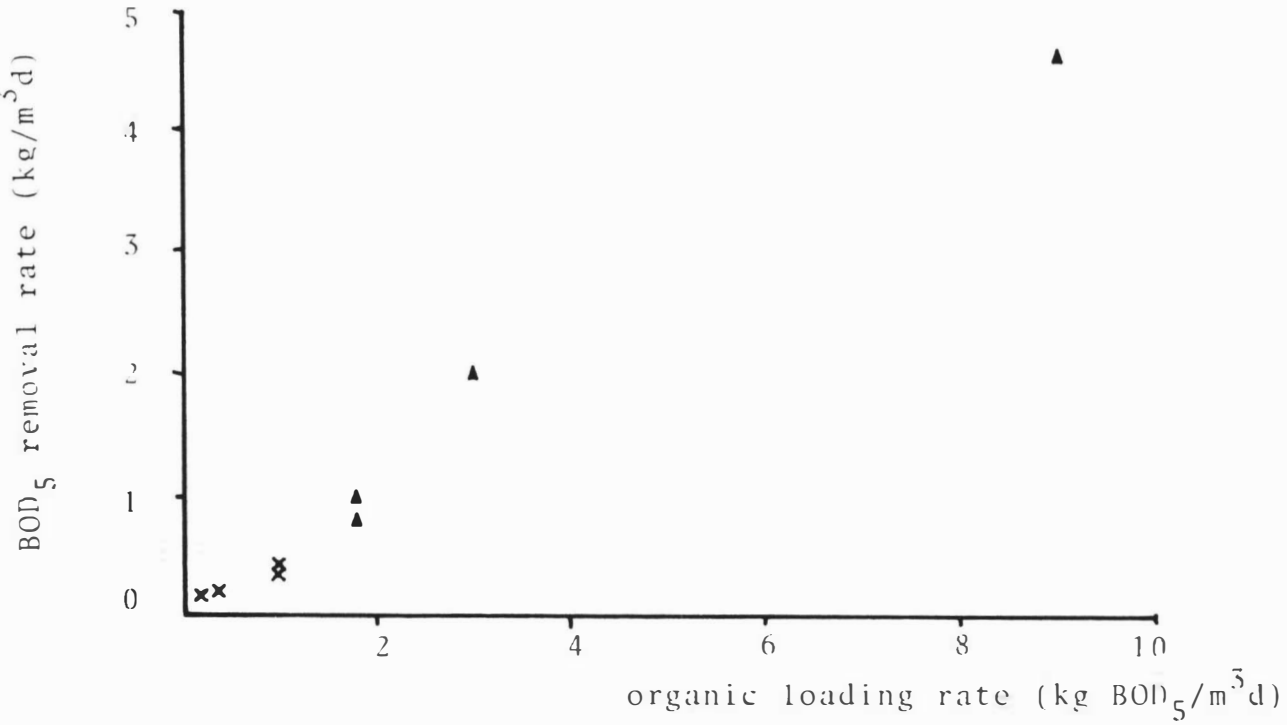


FIGURE 5.17: Relationship between applied BOD_5 and BOD_5 removal rate at different influent substrate concentraions

The data showed similar trends to those for the removal of COD as would be expected due to the high positive correlation between BOD and COD (r = 0.998). The BOD data was incomplete and in the Figure 5.16 there is far more spread in points than for the corresponding COD data (Figure 5.6). The wide discrepancy between duplicates in BOD_5 samples for the anaerobic filter effluent leads to little confidence being placed in this parameter.

Similar to the effluent COD, the effluent BOD_5 was solely dependant on the influent substrate concentration (Figure 5.13) and hydraulic retention time had little effect on this parameter.

The percentage BOD removal could not be related to organic loading rate with any degree of confidence due to the great spread in data points (Figure 5.14). A reduced percentage BOD_5 removal at increased organic loading rate was expected as was found in the COD data.

Regression models were not derived for the BOD data.

5.3 The Effects of the Experimental Factors on Gas and Methane Production

5.3.1 Gas Production

The influence of organic loading rate on total gas production is illustrated in Figure 5.18 with data points identified for both influent substrate concentration and hydraulic retention time.

Gas production was seen to increase with increasing organic loading rate; however the incremental increase in gas production decreased at higher influent substrate concentrations. For instance at 1000 mg BOD_5/ℓ, a reduction in retention time of 5 d to 1 d (which increased the loading rate 5 fold) resulted in a 4.9 fold increase in gas

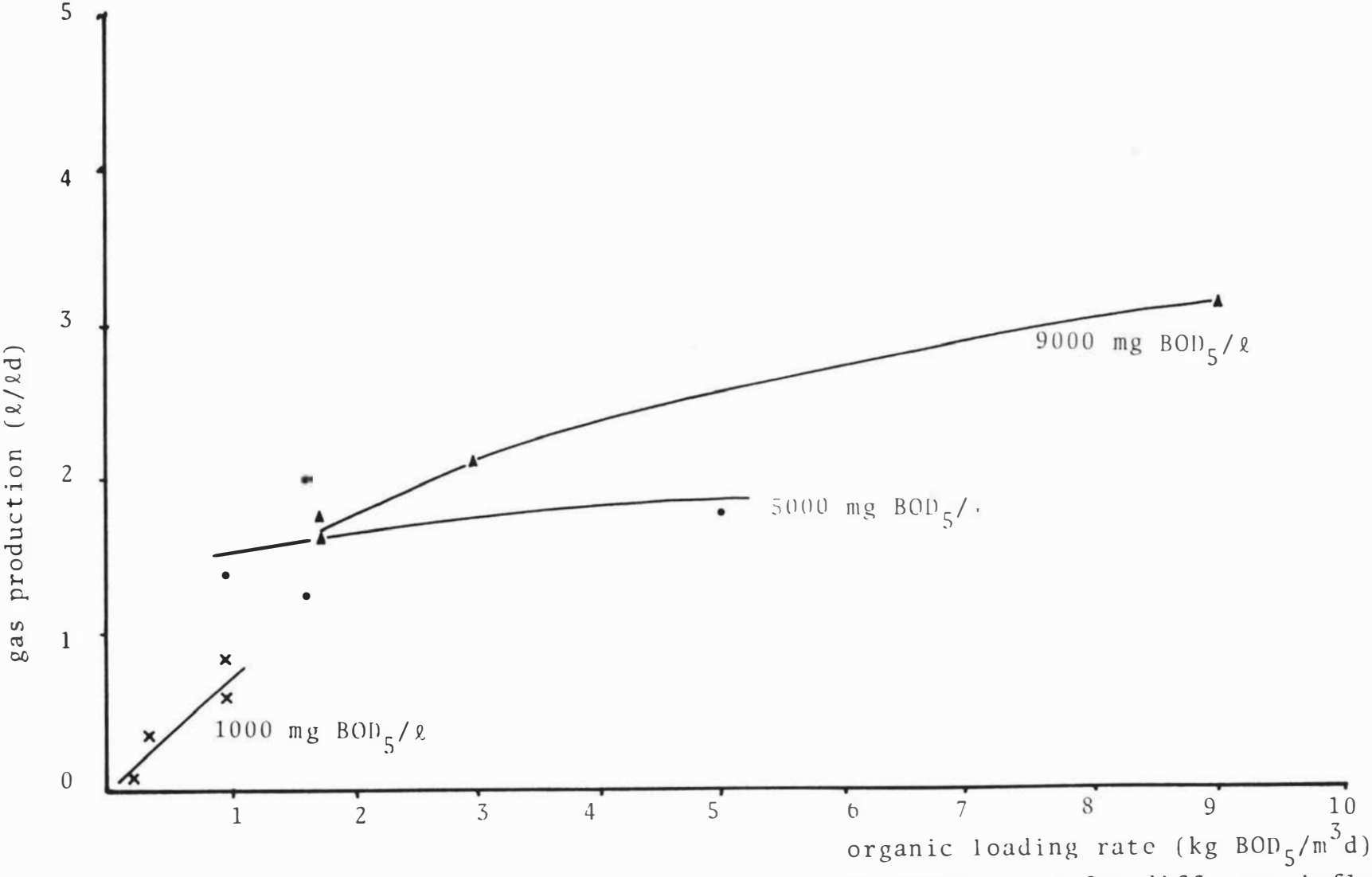


FIGURE 5.18: Total gas production as a function of organic loading rate for different influent substrate concentrations

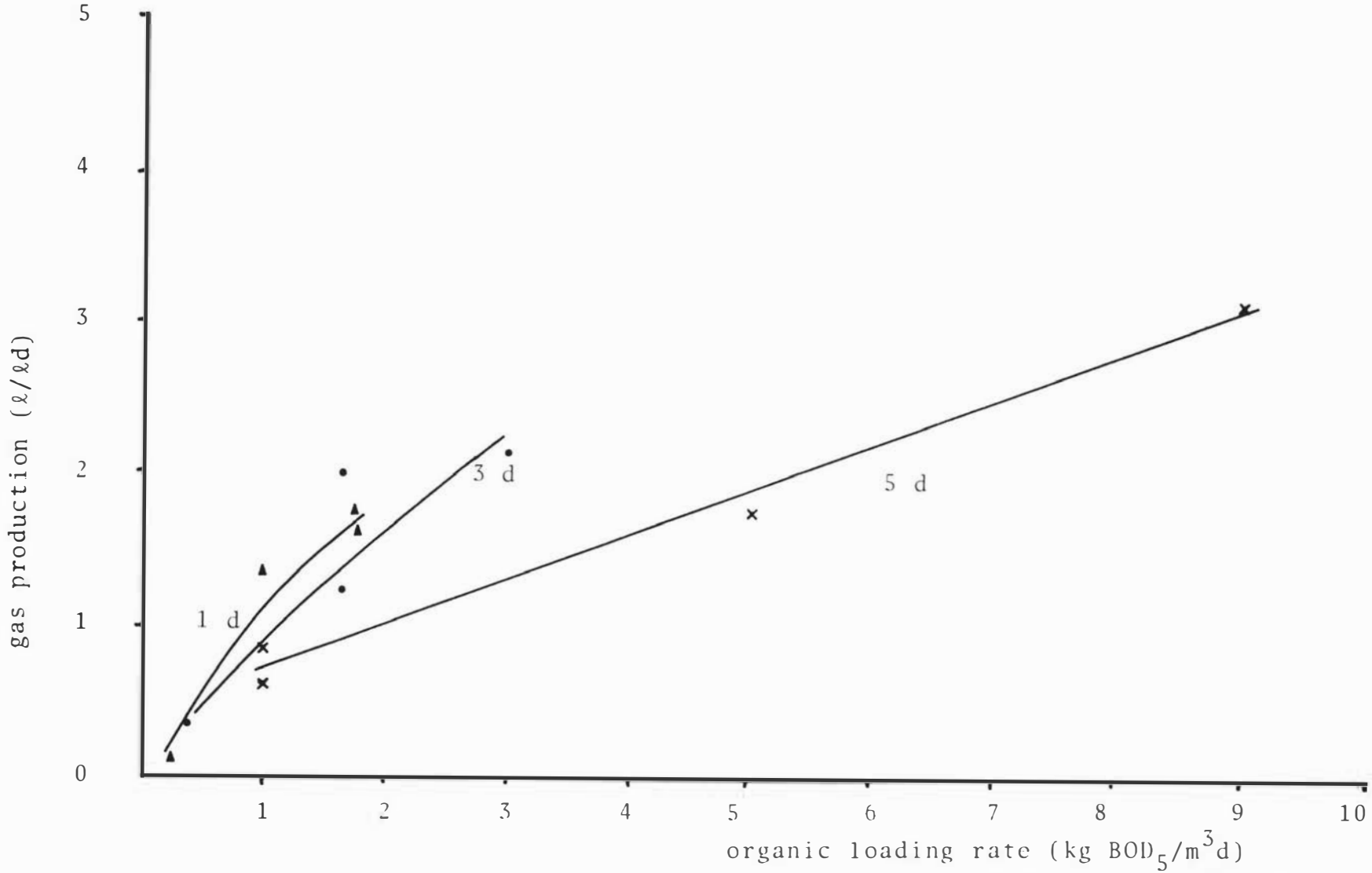


FIGURE 5.18a: Total gas production as a function of organic loading rate for different hydraulic retention times

production. However at 9000 mg BOD_5/ℓ a 500 percent increase in loading rate (run 12) resulted in only a 175 percent increase in gas production over run 9.

The effects of influent substrate concentration and hydraulic retention time are illustrated in Figures 5.19 and 5.20 respectively. Since the regression equation for total gas production includes a temperature term, some allowance must be made for this in the plots. In terms of amount of gas production involved, it is a small amount (0.23 ℓ/ℓ d). However it does represent some 10 percent of the range of values measured.

There is considerable scatter in this data set particularly at the mid points of both factors. The two runs carried out at 5000 mg BOD_5/ℓ & 3 d had a greater gas production at the higher temperature run (run 7). But this was also the run on the larger anaerobic filter so the temperature effect is not distinguishable by a cursory examination.

5.3.2 Methane Production

Figures 5.21 to 5.23 illustrate the effects of influent substrate concentration, hydraulic retention time and organic loading rate on production of methane by the anaerobic filters. It was found that there was an almost linear increase in methane production with both increasing influent BOD_5 (Figure 5.22) and decreasing retention time (Figure 5.23). However there was no marked increase in methane production at the highest loading rate as there was in the total gas production (Figures 5.19 and 5.20).

At feed concentrations less than 5000 mg BOD_5/ℓ, there was a greater incremental increase in methane production with increase in feed concentration than above that level. The slope of the curves for each hydraulic retention time in Figure 5.22 above 5000 mg/ℓ were approximately half

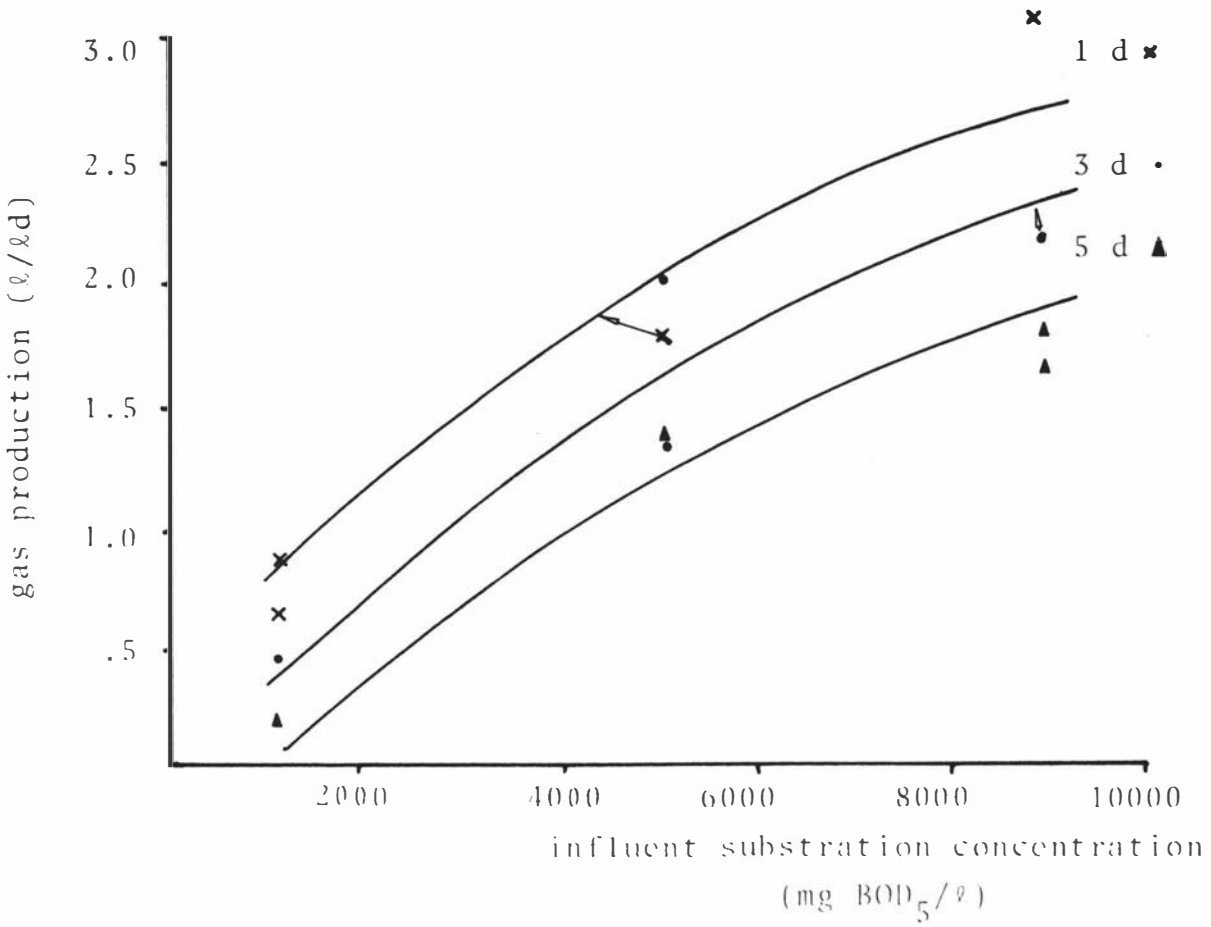


FIGURE 5.19: Effect of influent substrate concentration on gas production at different HRT

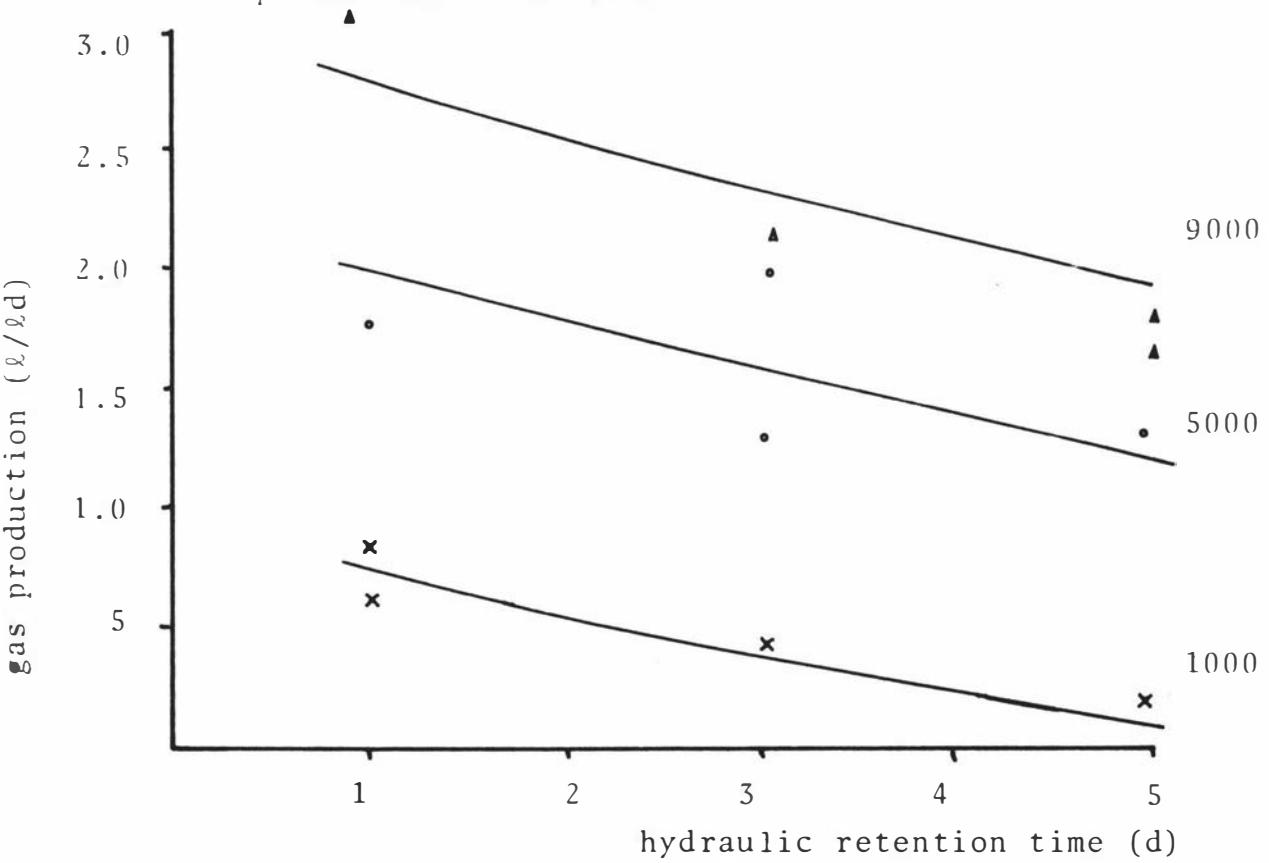


FIGURE 5.20: Effect of hydraulic retention time on gas production at different influent substrate concentrations

those in the range 0 to 5000 mg/ ℓ.

With respect to organic loading rate (Figure 5.21), a nine fold increase from 0.2 to 1.8 kg BOD_5/m^3d resulted in the methane production increasing 8.7 times from 0.14 ℓ/ℓd to 1.2 ℓ/ℓd. However, a further 5 fold increase in loading to 9.0 kg BOD_5/m^3d returned only a 1.3 fold increase in methane production. At 1.0 kg BOD_5/m^3d, there was a higher methane production at the higher feed concentration and longer retention time than at the converse combination.

5.3.3 Methane Content of the Digester Gas

The effect of organic loading rate on the percentage of methane measured in the digester gases is shown in Figure 5.24 and data points are identified for influent substrate concentration and hydraulic retention times.

Although Figure 5.24 indicates that there was a trend towards lower methane content of the digester gas at increased organic loading rate, there is considerable spread in the data. As parametric plots for both influent BOD_5 and hydraulic retention times, however, definite patterns emerged. For constant influent substrate concentration levels of 1000 and 5000 mg/ℓ (Figure 5.25), there was a small increase in percentage of methane in the gas as organic loading rate was increased. But at the highest substrate concentration of 9000 mg $BOD_5/ℓ$, the trend reversed and a dramatic drop in percent methane was evident as the loading rate was increased by decreasing the hydraulic retention time. The constant loading rate runs at 1 kg BOD_5/m^3d showed that higher levels of methane in the total gas were associated with lower levels of influent substrate concentration and shorter hydraulic retention times. The difference was considerable being from 85.1 percent methane at 1000 mg $BOD_5/ℓ$ and 1 d HRT, to 67.9 percent methane at 5000 mg $BOD_5/ℓ$ and 5 d HRT.

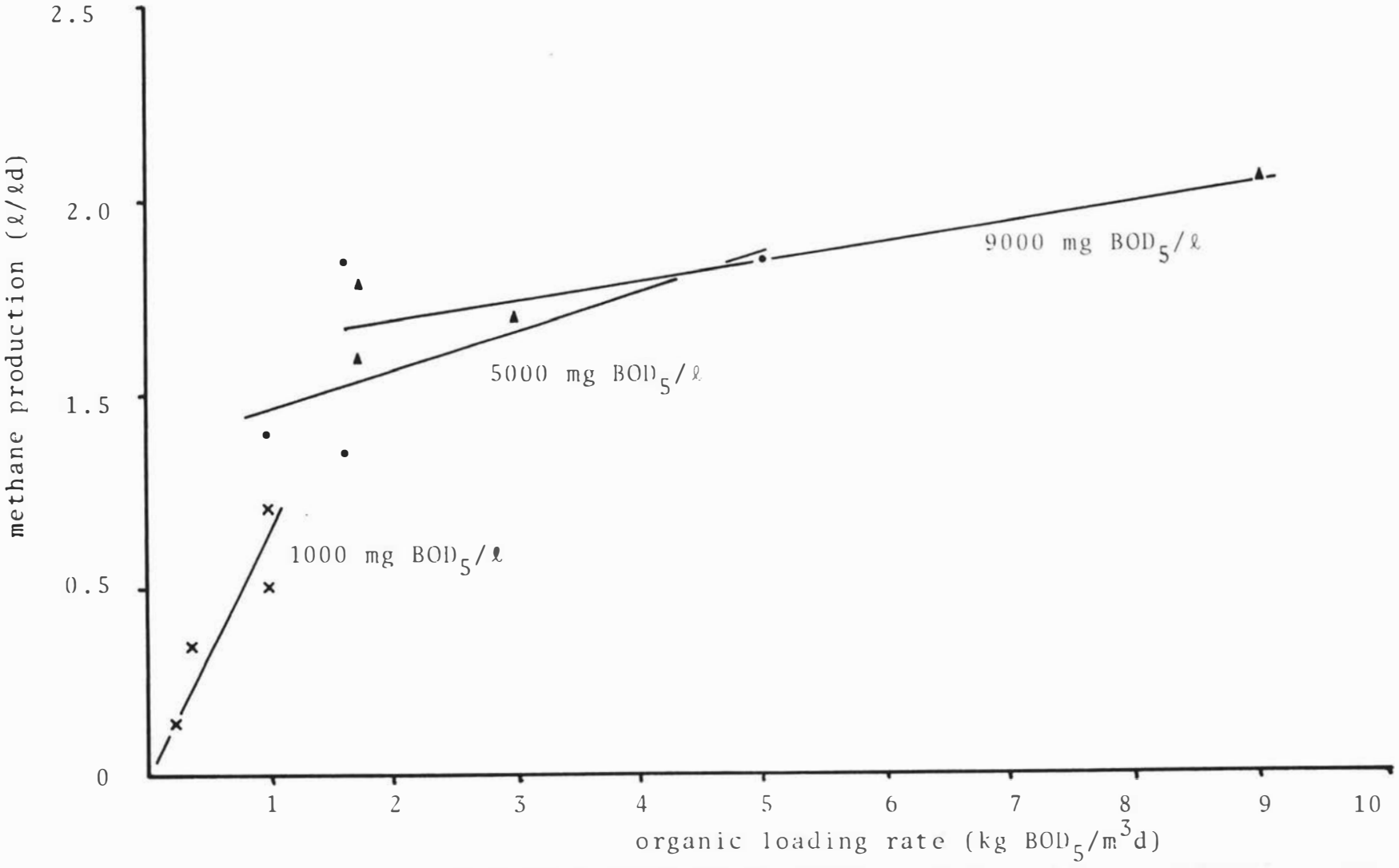


FIGURE 5.21: Methane production as a function of organic loading rate at different influent substrate concentrations

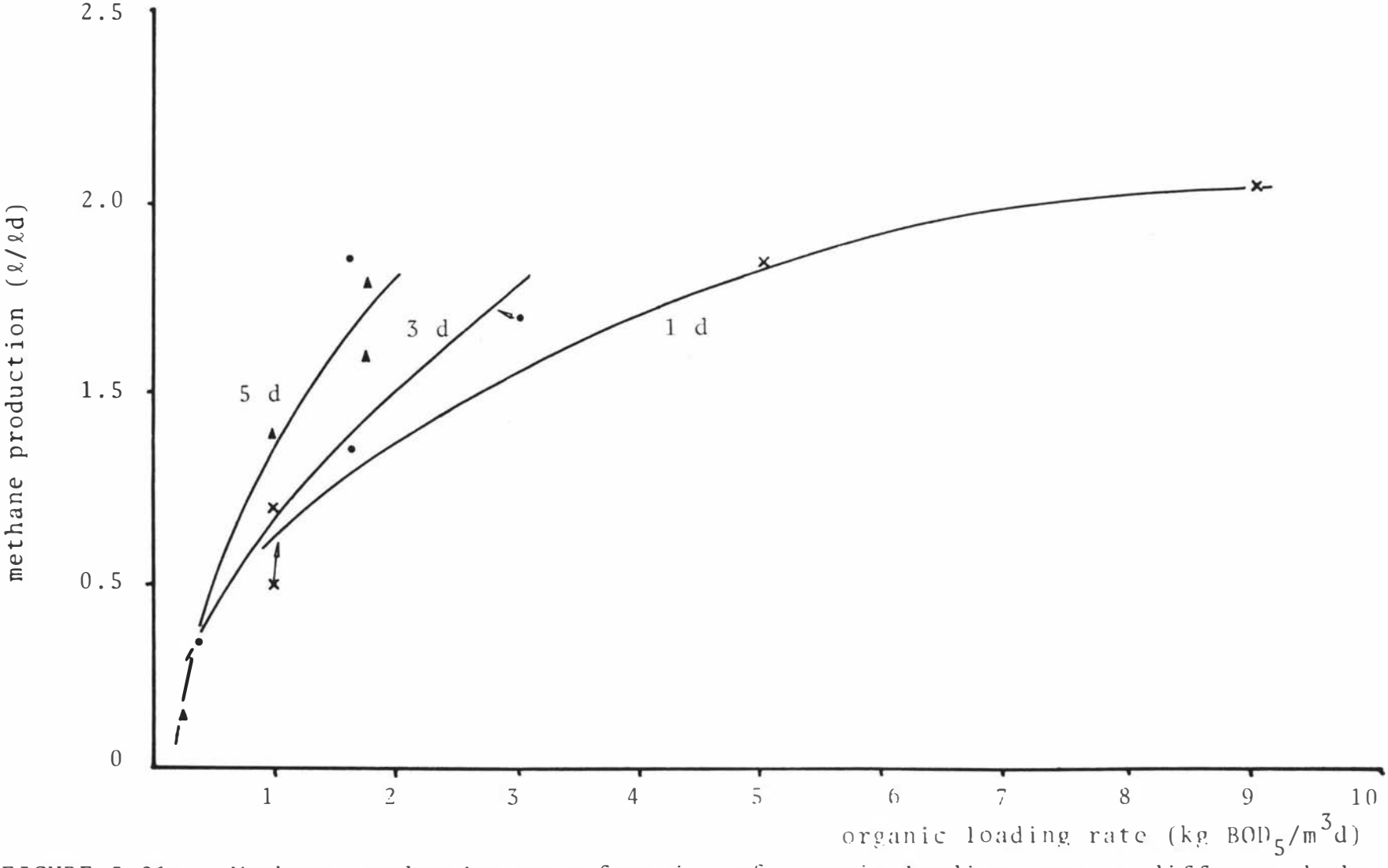


FIGURE 5.21a: Methane production as a function of organic loading rate at different hydraulic retention times

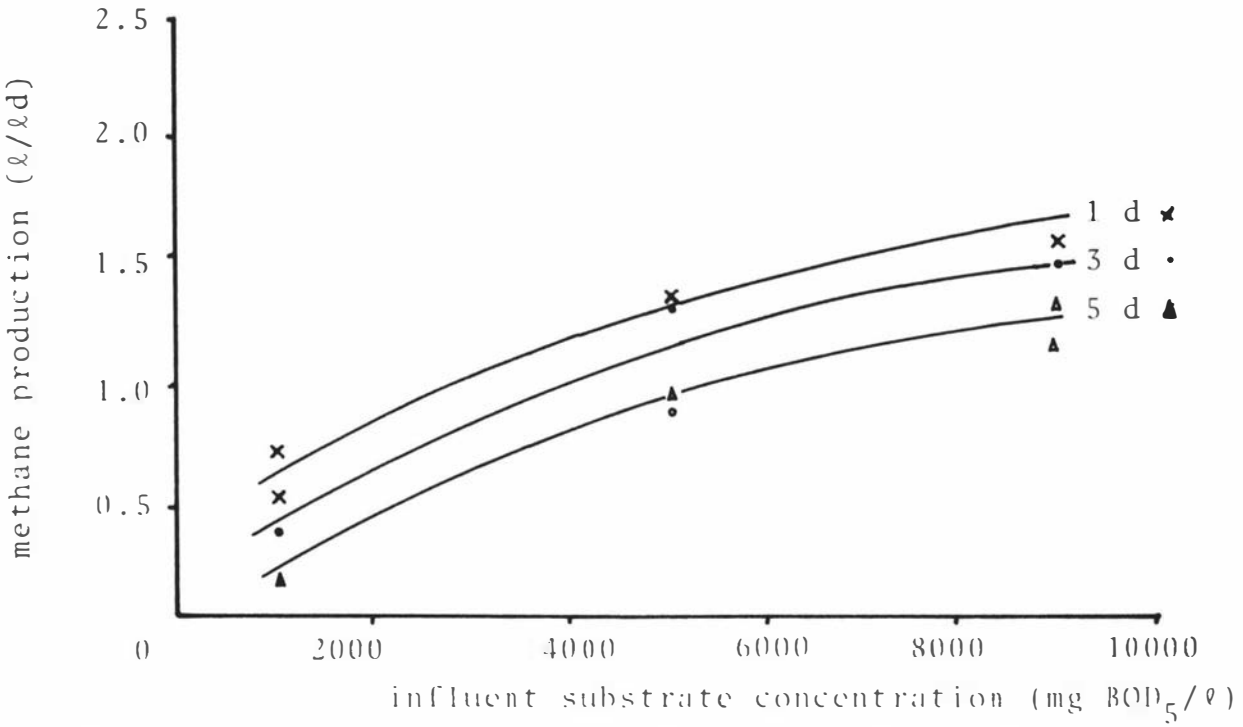


FIGURE 5.22: Effect of influent concentration on methane production at different HRT

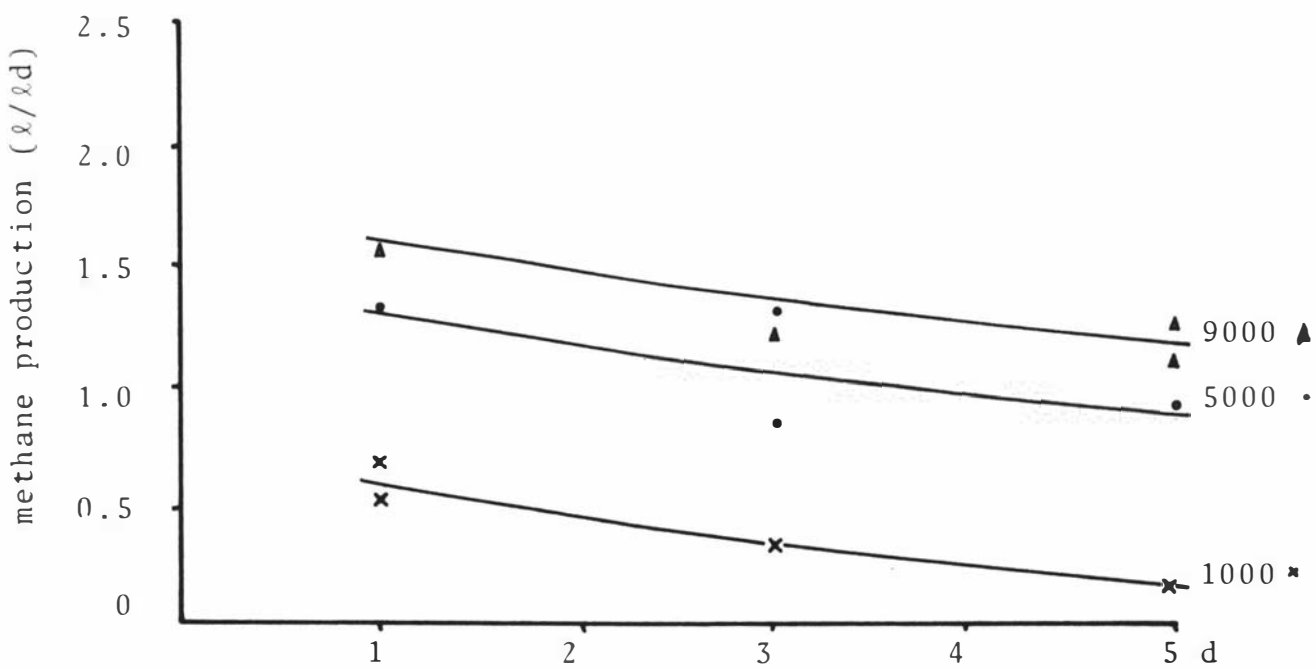


FIGURE 5.23: Effect of hydraulic retention time on methane production at different influent substrate concentrations

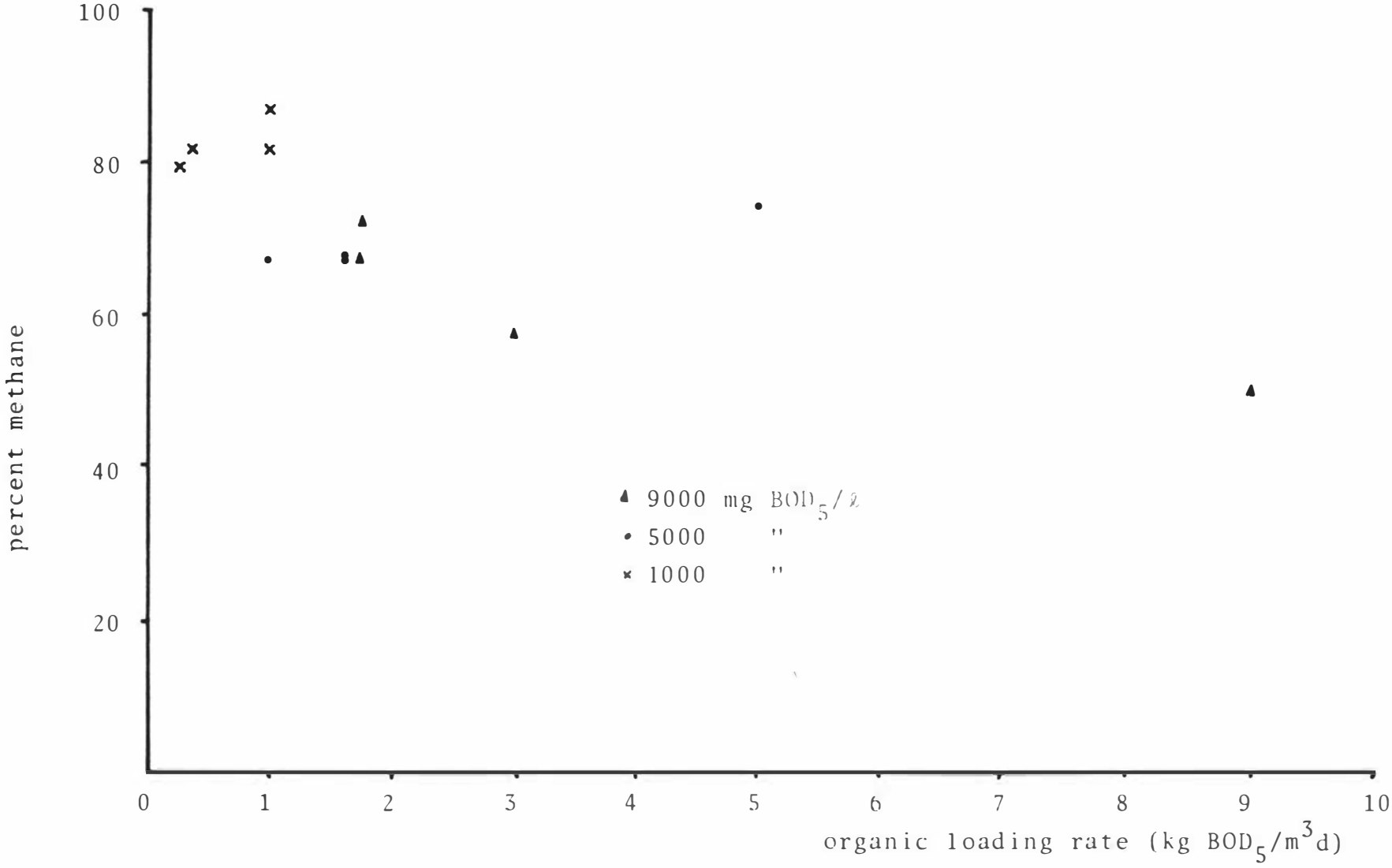


FIGURE 5.24: Methane content of digestor gas as a function of organic loading rate at different influent substrate concentrations

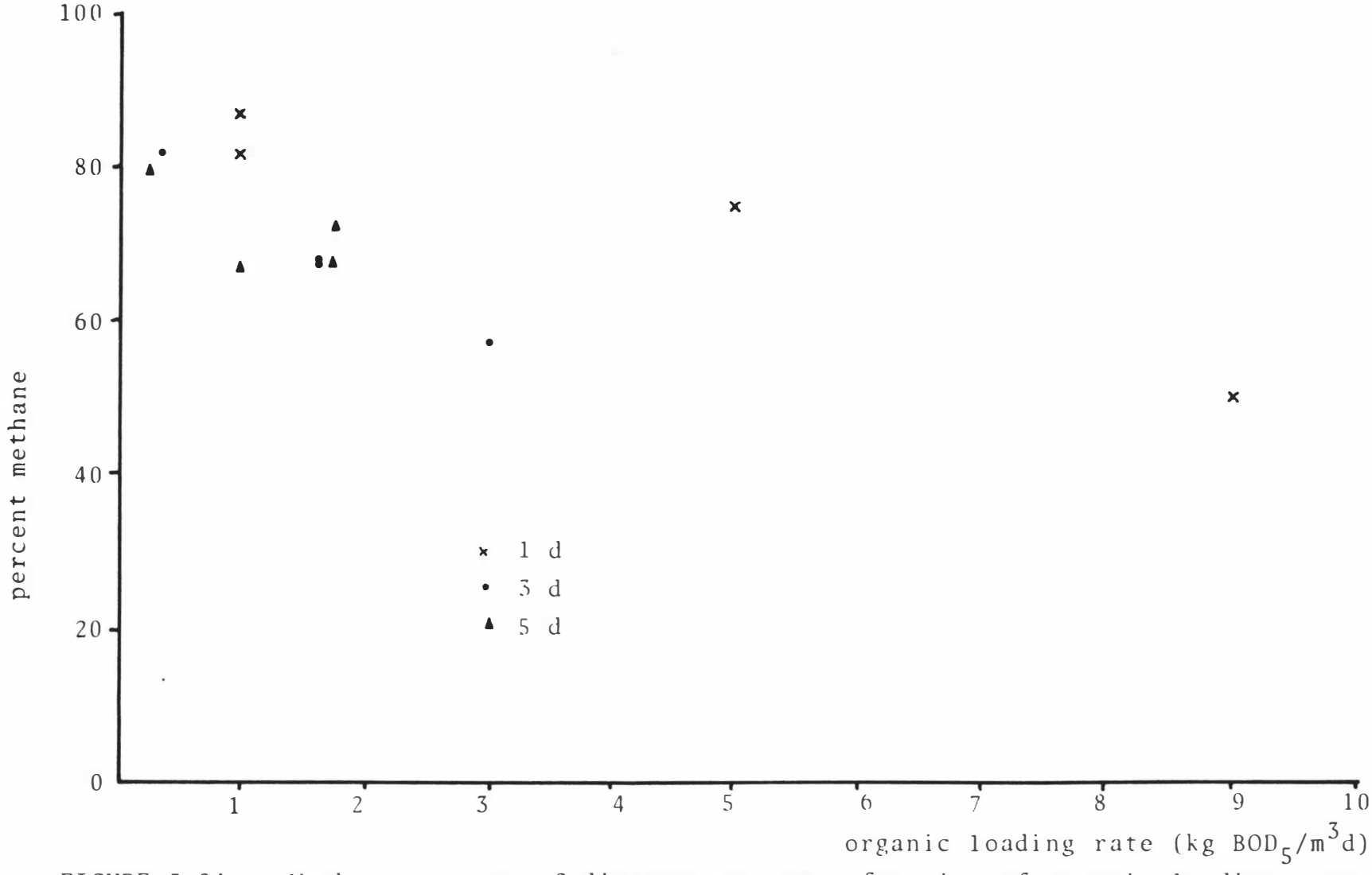


FIGURE 5.24a: Methane content of digestor gas as a function of organic loading rate at different HRT

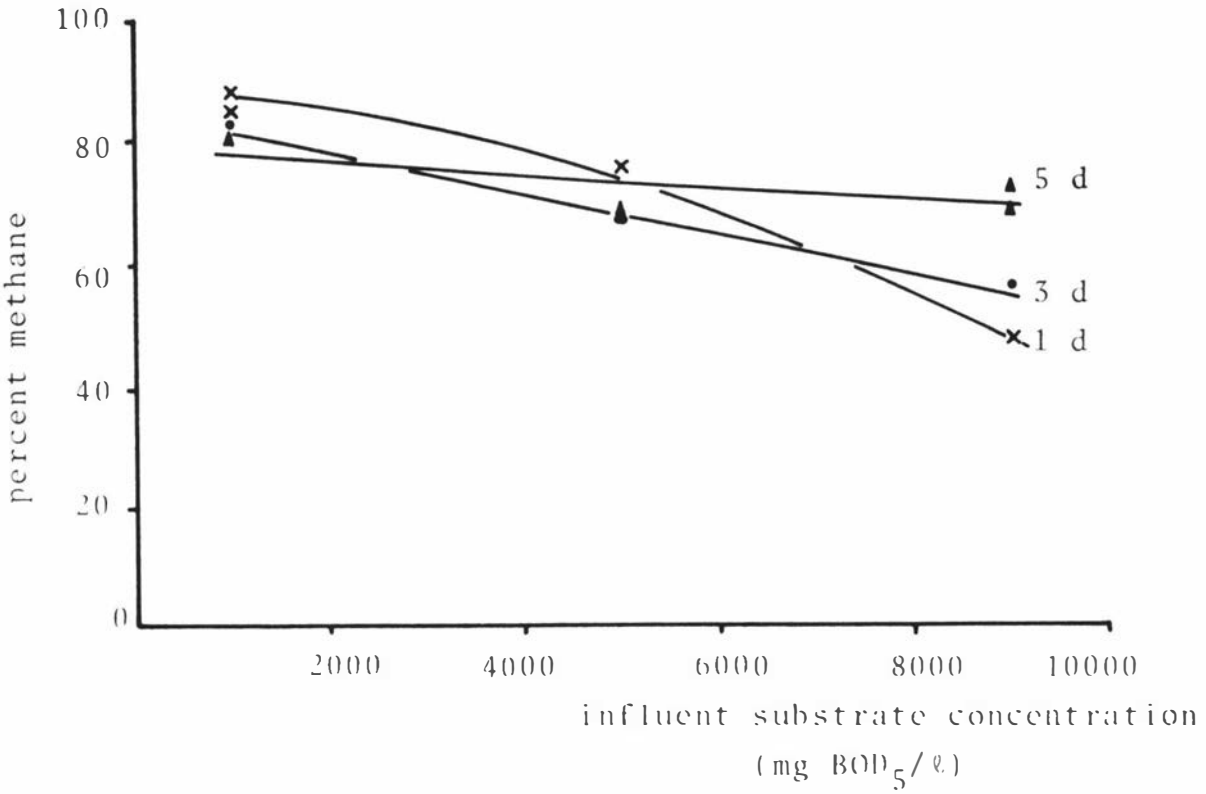


FIGURE 5.25: Effect of influent substrate concentration on methane content of digestor gas at different HRT

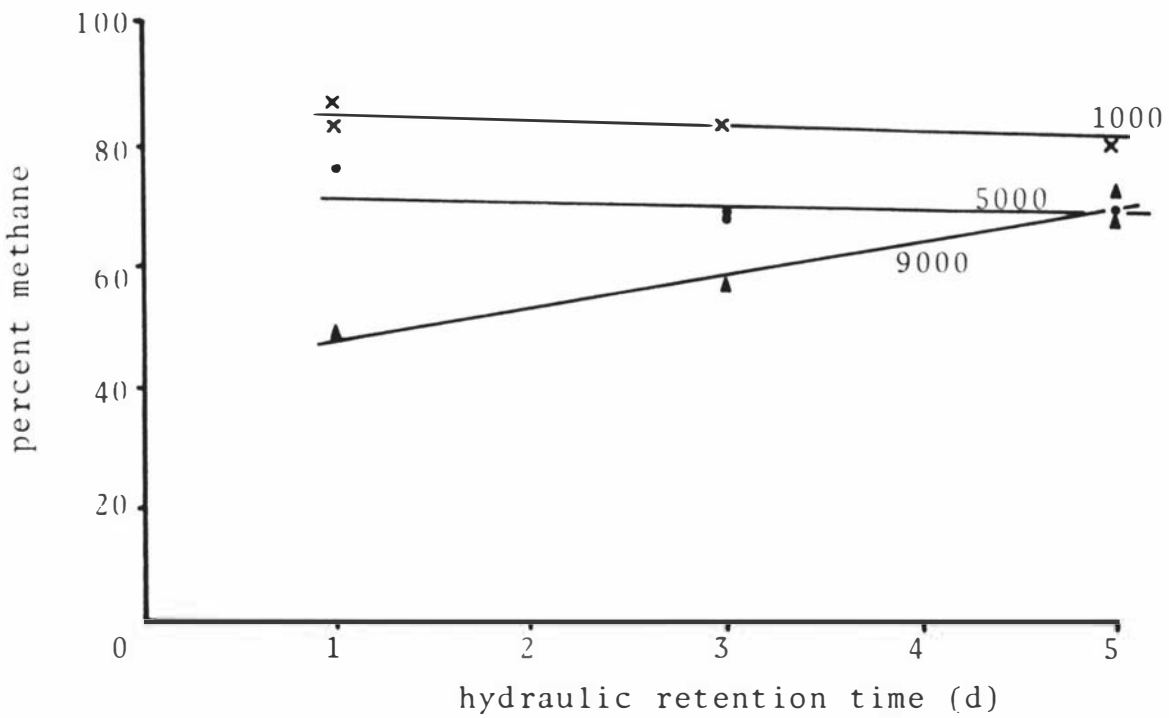


FIGURE 5.26: Effect of HRT on methane content of digestor gas at different influent substrate concentrations

5.4 The Effects of the Experimental Factors on pH, Alkalinity, Ammonia and Sulphide Concentrations

5.4.1 Effluent pH

Neither influent substrate concentration, hydraulic retention time or organic loading rate exerted any effect on filter effluent pH as can be seen from Figures 5.27 and 5.28. A narrow range of effluent pH between the values pH 6.75 and 7.25 was observed over the range of independant variables tested. This observed range was within the optimum range presented by McCarty (1964) for methanogenic bacteria.

Although the influent pH varied from run to run, this was not an independant variable. Influent pH was largely determined by the amount of $NaHCO_3$ that was added to the feed solution to maintain the filter pH at a favourable level. Also when steady state conditions were assumed to have been achieved a certain amount of "weaning" was carried out as time permitted. In most cases, the amount of buffer used could probably be reduced and the filter allowed to maintain its pH through retention of alkalinity.

5.4.2 Volatile Acid Concentration

Volatile acid concentration was found to be closely related to the organic loading rate (Figure 5.29). At loading rates of 0.2 to 1.0 kg BOD_5/m^3d, total volatile acid concentrations of less than 1000 mg/ℓ were observed. There followed a sharp increase in acid levels when the system was loaded above a level of 1.67 kg BOD_5/m^3d, with acid concentrations ranging from approximately 2000 mg/ℓ up to 4846 mg/ℓ at a loading rate of 9 kg BOD_5/m^3d. There was a corresponding decrease in the conversion of COD to methane with increased organic loading rate and this trend will be discussed in the next chapter. Individual volatile acids, measured for each run are presented in able 5.4. The higher total volatile acid concentra-

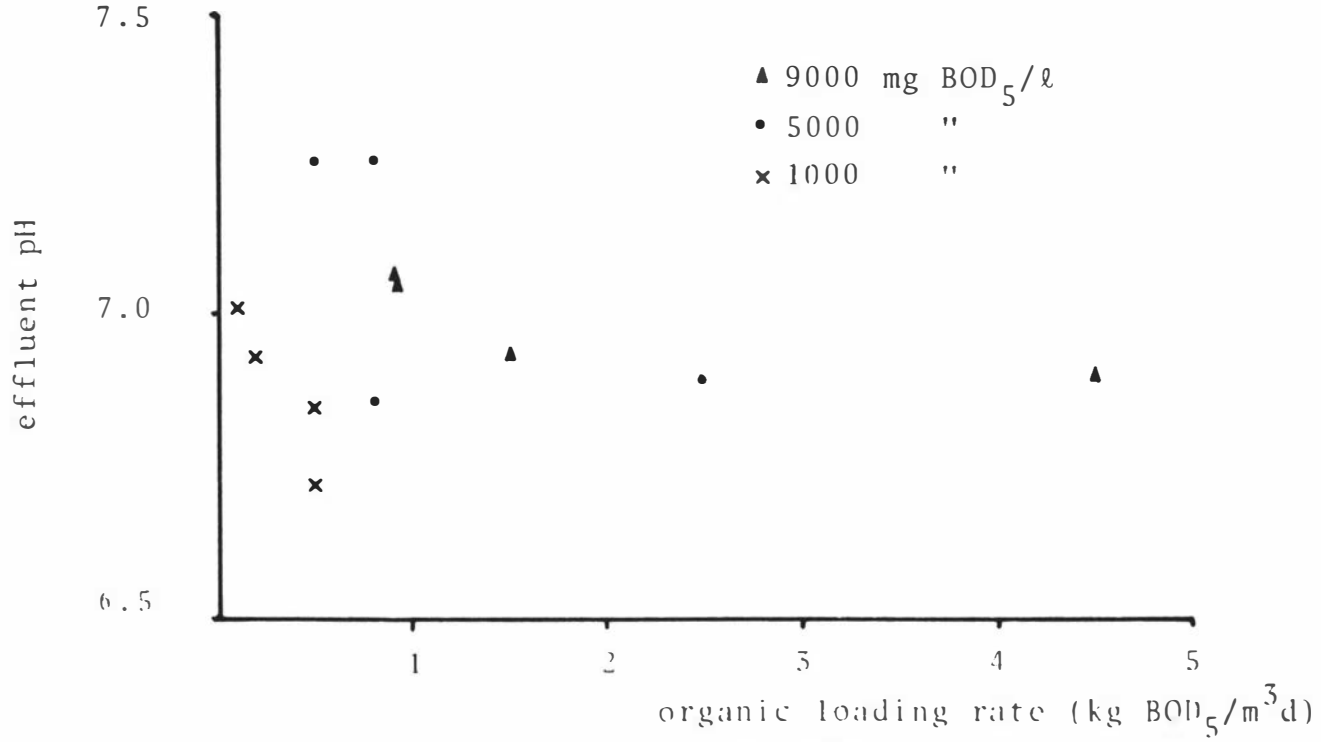


FIGURE 5.27: Effluent pH as a function of organic loading rate at different influent substrate concentrations

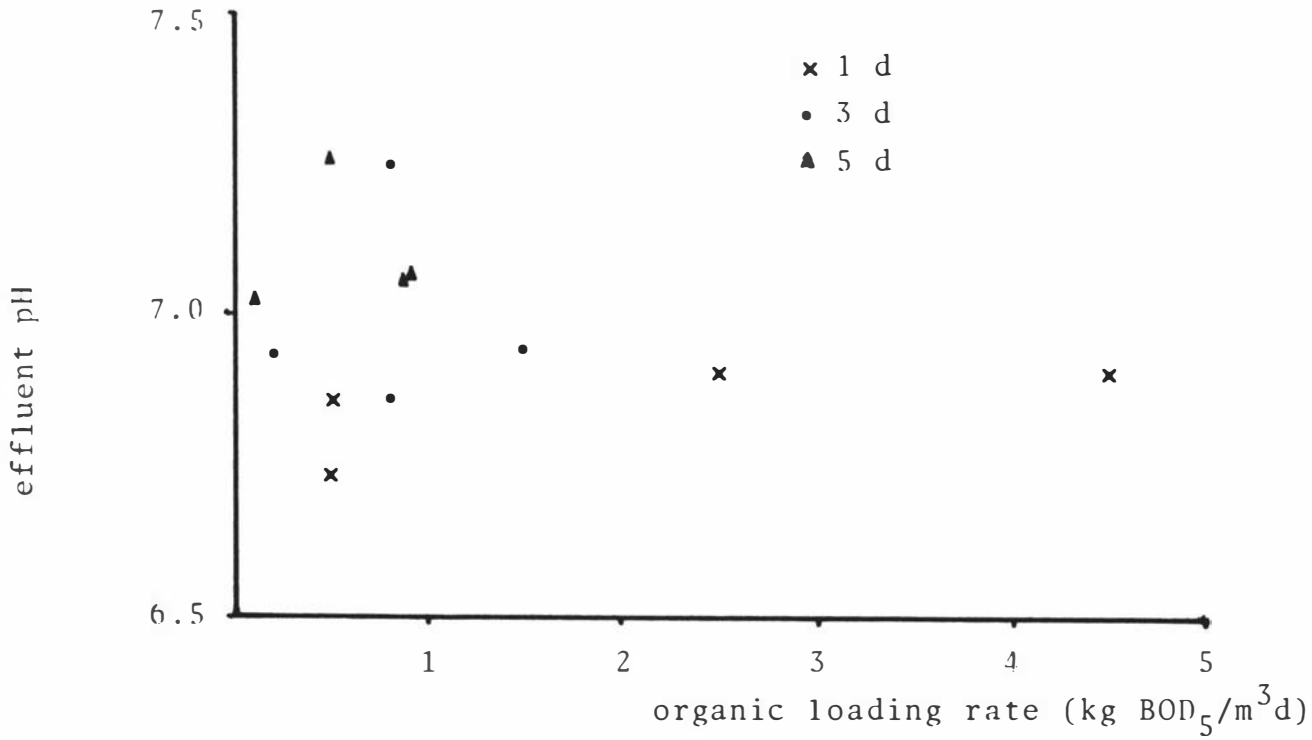


FIGURE 5.28: Effluent pH as a function of organic loading rate at different HRT

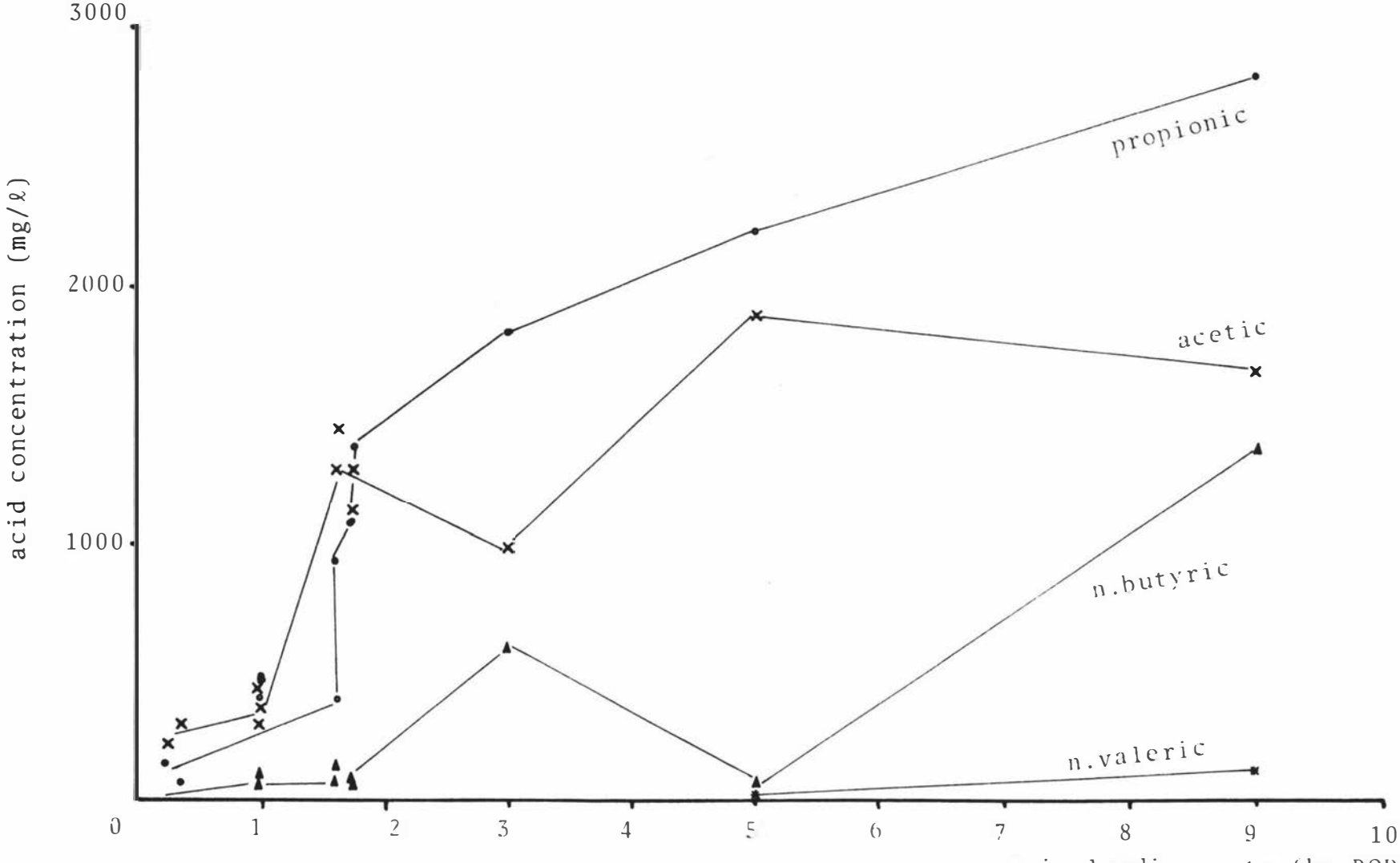


FIGURE 5.29: Individual volatile acids concentrations as a function of organic loading rate

TABLE 5.4: Individual Volatile Acid Concentrations Measured at Steady State Conditions. (All values in mg/ℓ)

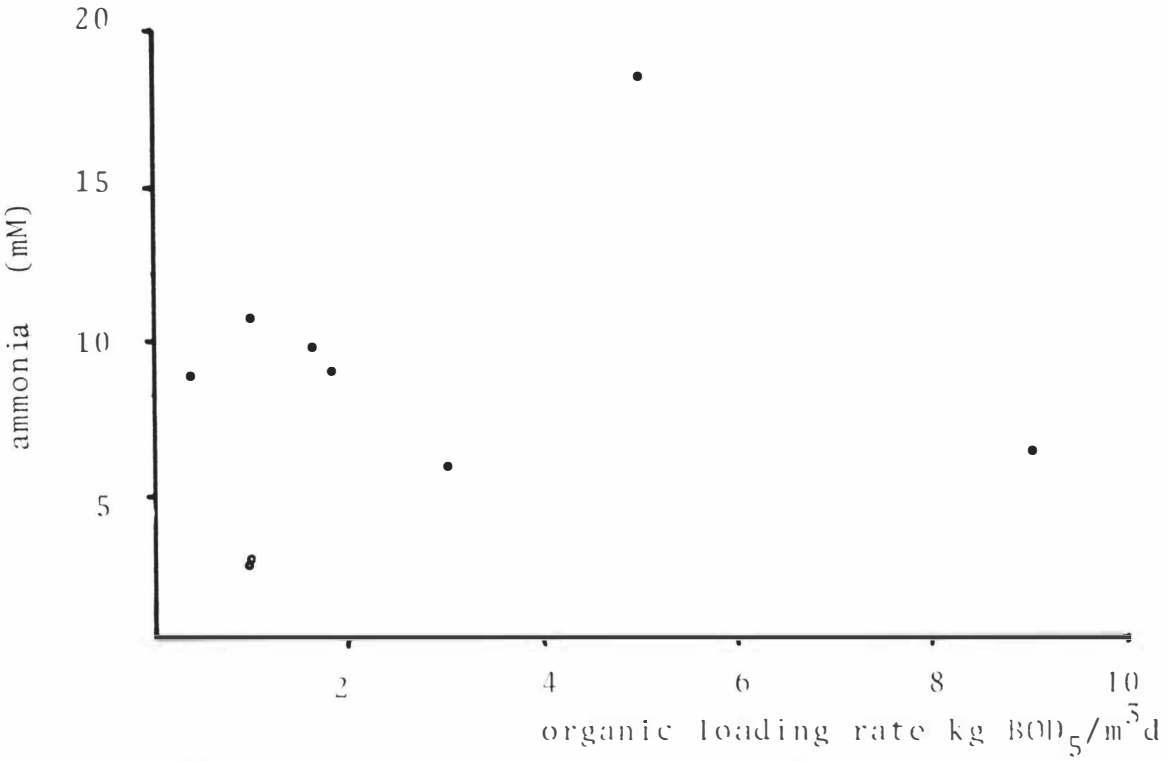
Run	HRT	Organic Loading Rate kg BOD_5/m^3d	Acetic	Propionic	iso-Butyric	Butyric	iso-Valeric	Valeric
1	1	1.0	378	414	-	tr	-	-
2	1	1.0	294	424	-	50	-	-
3	3	0.33	301	50	-	-	-	-
4	5	0.20	239	186	-	-	-	-
5	1	5.0	1872	2199	tr	77.2	tr	50
6	3	1.67	1441	348	-	128	-	-
7	3	1.67	1310	899	-	84	-	-
8	5	1.0	480	371	-	107	-	-
9	1	9.0	1626	2766	tr	1354	tr	90
10	3	3.0	965	1823	tr	586	-	-
11	5	1.8	1120	1047	-	75	-	-
12	5	1.8	1254	1356	tr	96.5	-	-

tr = trace quantity

tions were characterised by increased concentrations of higher organic acids. Butyric acid concentration increased from zero at the lowest total acid concentrations (and lowest organic loading rates of 0.2 and 0.33 kg BOD_5/m^3d), to 1354 mg/ℓ at the highest total acid and loading rate. n-valeric acid was detected in measurable concentrations at loading rates of 5.0 and 9.0 kg BOD_5/m^3d.

5.4.3 Ammonia Levels

There was a slight positive correlation between filter effluent ammonia concentrations and organic loading rate (Figure 5.30). When one considers that influent total nitrogen concentration is directly proportional to influent substrate concentration (Table 5.2), it is interesting to note that at the 5000 mg $BOD_5/ℓ$ and 9000 mg $BOD_5/ℓ$ influent substrate concentration, the effluent ammonia concentration was generally higher at the lower of these two influent substrate concentrations. At a constant level of influent substrate concentration, one would expect higher effluent ammonia concentrations as hydraulic retention time was reduced due to incorporation of the nitrogen into new cell growth, there being less conversion of COD to methane at the shorter hydraulic retention time. But there was too wide a scatter in the data to state any such relationship with confidence.



RE 5.30: Filter effluent ammonia concentration as a function of organic loading rate

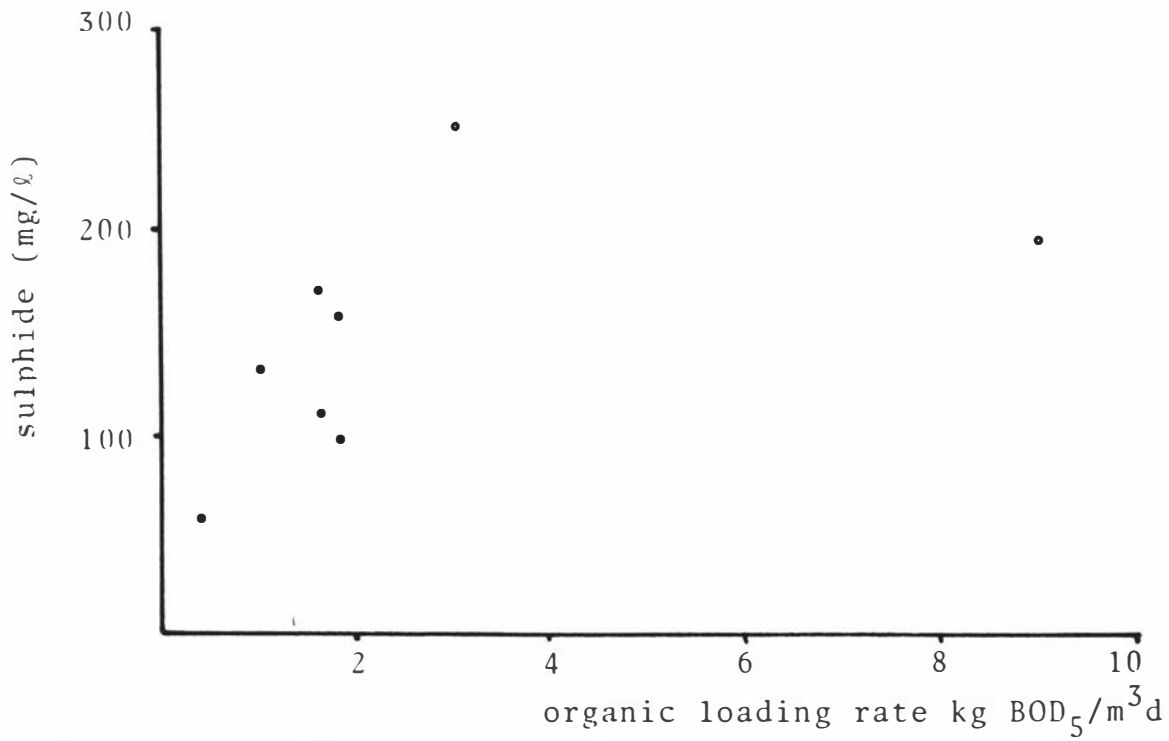


FIGURE 5.31: Filter effluent sulphide concentration as a function of organic loading rate

5.4.4 Sulphide Concentrations in the Filter Effluent

Total sulphide concentration in the filter effluent was related to organic loading rate (Figure 5.31). There was a near linear increase in sulphide concentration as the organic loading rate was increased to 3 kg BOD_5/m^3d and a slight decrease at 9 kg BOD_5/m^3d. This increase was correlated to the increase in influent substrate concentration and so there appears to be a positive relationship between influent sulphate level and effluent total sulphide concentration.

The sulphide concentrations in many runs were above the generally accepted level of strong inhibition by sulphide of 150 mg/ℓ. During the experimental design runs, no attempt was made to reduce this high sulphide concentration as conditions had to be kept constant throughout the design to reduce the effects of uncontrollable variables. However, after the completion of the design and analysis of the data, two further runs were carried out, one on each filter unit, under identical conditions except for the addition of $FeCl_3.9H_2O$ at 2.5 g/ℓ to the feed of AF1 to precipitate the sulphide out of the filter contents. The data from these runs is presented in Table 5.5 along with the previous run from the experimental design for comparison. A Wald-Walfowitz Runs Test (Lipson and Sheth, 1973) on the data indicated that there was no significant difference at 95% confidence level, between the groups of data.

5.4.5 Alkalinity

As in the case of effluent volatile acid concentration, there was significant (at 99 percent confidence level) correlation between alkalinity and effluent COD. In consequence, alkalinity exhibited similar response to effluent COD and its concentration was entirely dependant on that quantity. Figure 5.32 illustrates this similar pattern.

TABLE 5.5: Data from the Repeat Experimental Runs to Assess the Effect of Sulphide Level on Filter Performance

	Parameter	Design run	Repeat run 1	Repeat run 2
input	Feed conc.	9000 mg BOD/ℓ	9000 mg/ℓ	9000 mg BOD/ℓ
input	Hydraulic retention time	1d	1d	1d
input	Temperature	35°C	35°C	35°C
input	AF unit	1	1	2
input	Feed COD	47175 mg/ℓ	46382 mg/ℓ	46382 mg/ℓ
	gas production	3.110 ℓ/ℓd	3.247 ℓ/ℓd	2.865 ℓ/ℓd
	methanein gas (%)	49.9	47.8	46.4
effluent	pH	6.90	6.95	6.90
effluent	COD	31090 mg/ℓ	30010	30795
effluent	BOD	4455 mg/ℓ	4230	4285
effluent	ammonia	6.6 mM	8.2 mM	9.6 mM
effluent	sulphide	<50 mg/ℓ	18.6	293
effluent	alkalinity (as $CaCO_3$)	6860 mg/ℓ	5995	5720
effluent	suspended solids	740 mg/ℓ	555	230
effluent	volatile acidity (as acetic)	4846 mg/ℓ	3735	4163
	COD removed (%)	34.1	35.3	33.6
	BOD removed (%)	50.5	53.0	52.6
	COD removal rate (kg/m^3d)	16.08	16.37	15.58
	FTCM	0.246	0.270	0.243

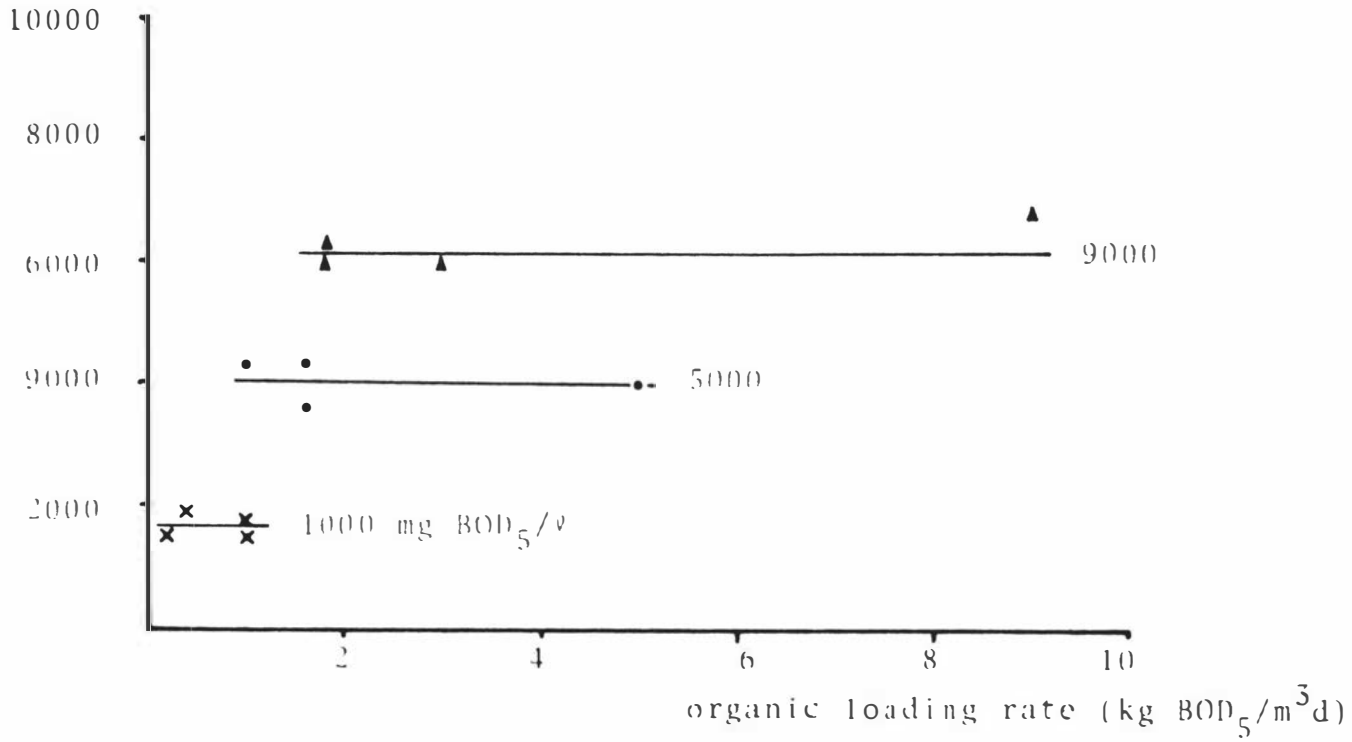


FIGURE 5.32: Effect of organic loading rate on filter alkalinity

5.5 The Effect of Filter Column Height

It was noted earlier in this study that recirculation of the filter effluent was practised. It was considered that such recirculation was necessary to reduce the possibility of potentially deleterious pH profiles vertically up the columns that could occur in a flow regime that included a large component of plug flow.

To ascertain the flow regime present in the anaerobic filter, three trials were carried out on the smaller filter prior to seeding and start up. The filter was filled with water and pumping started to establish the flow rate,

with and without recirculation, at the desired level.

At a time t = 0, a step change in the feed was introduced in the form of a solution of the dye safranin. The filter effluent was then monitored by measuring the absorbance at 530 nm (the approximate absorbance maximum for safranin); and the dye concentrations and times noted. The results are shown in Figure 5.32. The ordinate is the ratio of the concentration C of the dye at time t to the concentration of dye in the feed, C_o; the abscissa is a dimensionless time parameter Qt/V where:

Q = volumetric flow rate (ℓ/h)

t = time (h)

V = void volume of the filter (ℓ)

The curves on Figure 5.32 are known as the F curves Danckwerts (1953). Superimposed on the figure are the F curves for several ideal flow regimes, namely pure plug flow, fully developed laminar flow and a perfectly mixed reactor.

The trials carried out were at theoretical retention times (V/Q) of 10 d for the unrecycled filter and 1 and 5 d for the recycled filter. From the results it was apparent that a 10d unrecirculated regime exhibited near laminar flow characteristics and the changing slope in the region Qt/V = 0.5 to 0.75 could indicate some plug flow was present. A 10 d hydraulic retention time was not utilised in the filter study except under the start-up conditions.

The results from the 1 d and the 5 d recirculation retention times indicated that the anaerobic filter was essentially a completely mixed reactor under these conditions. This result meant that there should have been little or no difference between samples taken for analysis at the sampling points up the length of the larger column when it was operating; in fact this was the case.

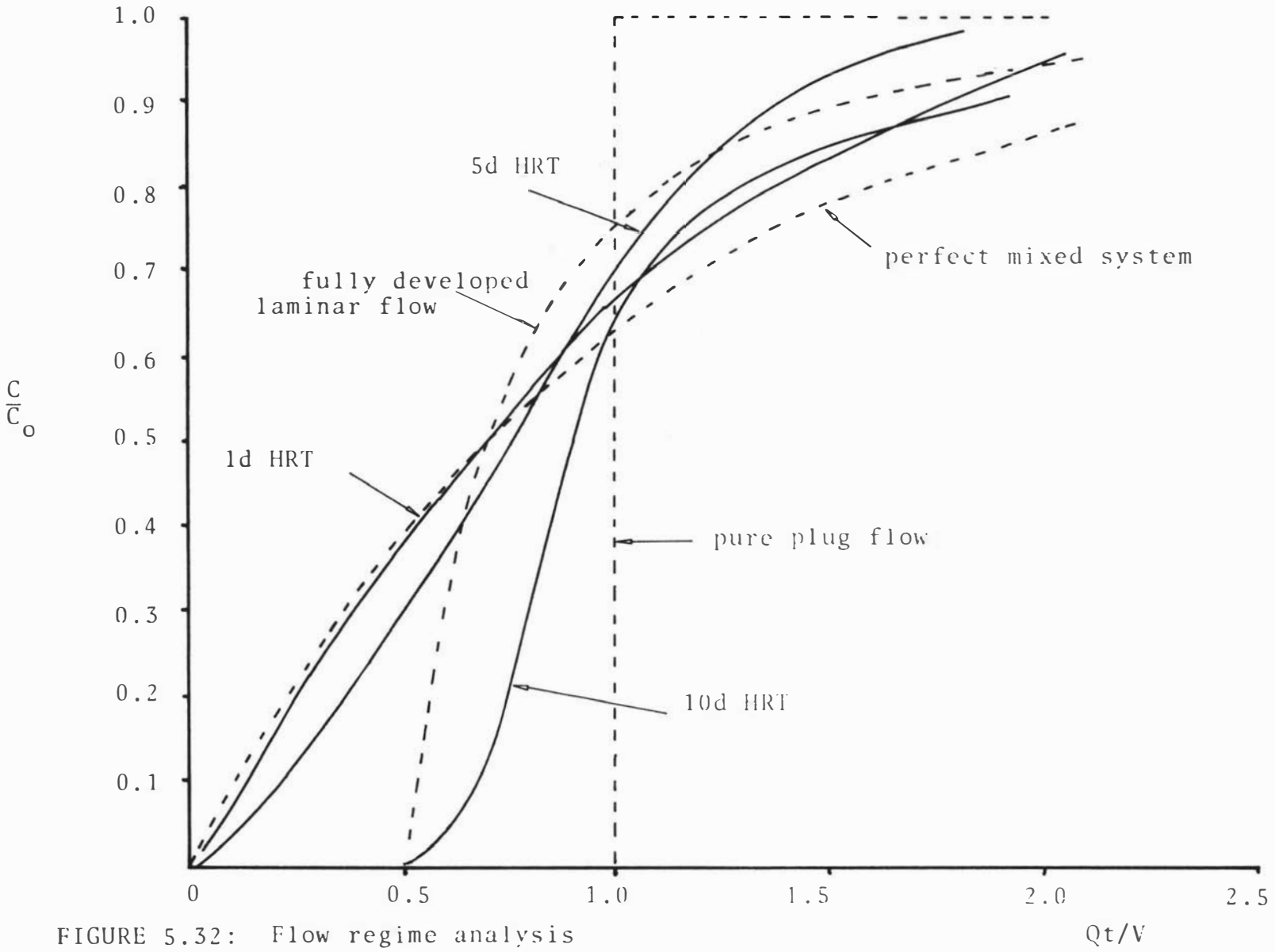


FIGURE 5.32: Flow regime analysis

In the following table (Table 5.5), the values for pH, COD (soluble) and ammonia are tabulated along with the corresponding filter effluent values for steady state conditions.

Although there is some variation between values up the column height, none appears to be attributable to the height parameter as the variation occurs at random.

It is therefore fair to say that the recirculated anaerobic filter was behaving as a completely mixed reactor.

TABLE 5.6: Steady State COD, pH and Ammonia Levels as a Function of Column Height (AF2 only)
5=top of column, 1=bottom

Run No.	2			3			5			7			11			12		
Level	COD	pH	NH_3	COD	pH	NH_3	COD	pH	NH_3	COD	pH	NH_3	COD	pH	NH_3	COD	pH	NH_3*
Effluent	1888	6.75	2.35	1931	6.95	9.15	17378	6.90	18.1	9879	7.25	9.7	27677	7.05	8.9	25508	7.05	
5	1841	6.70	2.3	2166	6.90	10.4	17527	6.80	17.3	10456	7.15	9.6	28010	7.15	7.7	25487	7.05	
4	2045	6.75	2.3	1956	6.95	10.6	16790	6.75	17.8	9514	7.30	10.4	27163	6.90	10.4	24860	7.00	
3	1787	6.70	2.4	1954	6.85	9.8	17690	6.75	18.0	9725	7.25	11.7	28340	7.00	8.4	25770	7.05	
2	1876	6.70	2.45	1979	6.90	8.2	18755	6.75	17.0	9807	7.10	12.2	27464	7.05	10.0	25945	6.95	
1	2024	6.65	2.4	2042	6.90	9.5	18036	6.85	18.4	10040	7.10	9.3	27885	7.05	9.35	25490	7.00	
mean	1910	6.70	2.37	2004	6.90	9.6	17696	6.80	17.8	9903	7.20	10.5	27756	7.03	9.11	25510	7.01	
range(1)	13.5	1.5	6.3	11.7	1.4	23	10	2	8	9	3	24	4	3	26	4	1	

Units are: COD mg/ℓ; pH units; NH_3 mM

* NH_3 not measured run 12

1 as percent of mean

CHAPTER SIX

PERFORMANCE OF THE ANAEROBIC FILTERS

CHAPTER SIX

PERFORMANCE OF THE ANAEROBIC FILTERS

6.1 Introduction

The operation of the anaerobic filters used in this study generally proceeded smoothly once several, largely mechanical, problems were ironed out. These related mainly to the efficient operation of the pumps used. Initially, positive displacement piston pumps were used, however, these proved particularly unsatisfactory. The rapid accumulation of biological solids on the inlet and outlet ball valves rendered them inoperative. A switch to peristaltic action pumps solved this problem.

The only other mechanically controlled facet of the filter operation, was that of temperature. On several occasions, due to power failures, the filters were cooled to below the normal 30-35^{o}C operating temperature for periods of up to two days. This resulted in reduced gas production but it was re-established again within approximately twelve hours of restoring operating temperatures, thus proving the system to be tolerant of fluctuations in temperature. Young & McCarty (1967) in their initial anaerobic filter studies pointed out the benefits of operating an anaerobic digester at near ambient temperatures and, from the temperature tolerance shown by this study when good gasification rates were manifest during the temperature drops, a case could be made for ambient temperature operation for industrial wastes.

Municipal sewage, which is much more dilute than the wastes generally used for anaerobic studies, has been successfully treated by anaerobic filtration at ambient temperatures on a pilot scale (Genung *et al*, 1976). Within seasonal temperature variations of 10 to 26^{o}C an average of 50 percent BOD_5 removal was achieved.

A considerable problem associated with this study was the extended periods required to carry out a limited number of experimental trials. Unfortunately this is a function of the anaerobic process itself. In another study on the digestion of yeast plant effluent in stirred reactors, the time delays were considered a problem in testing organic loading rates over the range 3.28 to 11.35 kg COD/m^3d (Hansford & Richtor, 1975). In this study a range of 1.15 to 47.2 kg COD/m^3d was coped with, in order to assess the efficacy of the process over expected real conditions. The effects of the long term nature of the experimental trials are discussed in subsequent chapter. This section will be confined to the effects of the independent variables applied to the filter units and their responses.

6.2 The Treatment of High Strength Waste Water by Anaerobic Filtration

It was noted in Chapter 2 that a wide range of waste types have been applied to the anaerobic filter with varying degrees of treatment efficiency. It is therefore necessary that the scope of the present study be placed in that context. In Figure 6.1, the Figure 2.1 is reproduced, showing the primary variables of influent substrate concentration and hydraulic retention time of previous studies with those of the present study superimposed. From this figure it may be noted that three of the runs carried out in the present study are located in the area relatively well covered by previous studies and the other nine extend the range of influent substrate concentration up to almost 50,000 mg COD/ℓ. In this way, expected real effluent concentration strengths could be covered, which would involve loading rates on a process system higher than previously tried, as well as those already documented and the whole analysed statistically via a rational model. In this way it was hoped that the performance of the anaerobic filter over a wide range of conditions could be described with reasonable accuracy in terms of a few input parameters.

This upper range of influent substrate concentration is of particular importance in terms of the on-site treatment of high strength industrial wastes with little or no adulteration, including dilution with lower strength

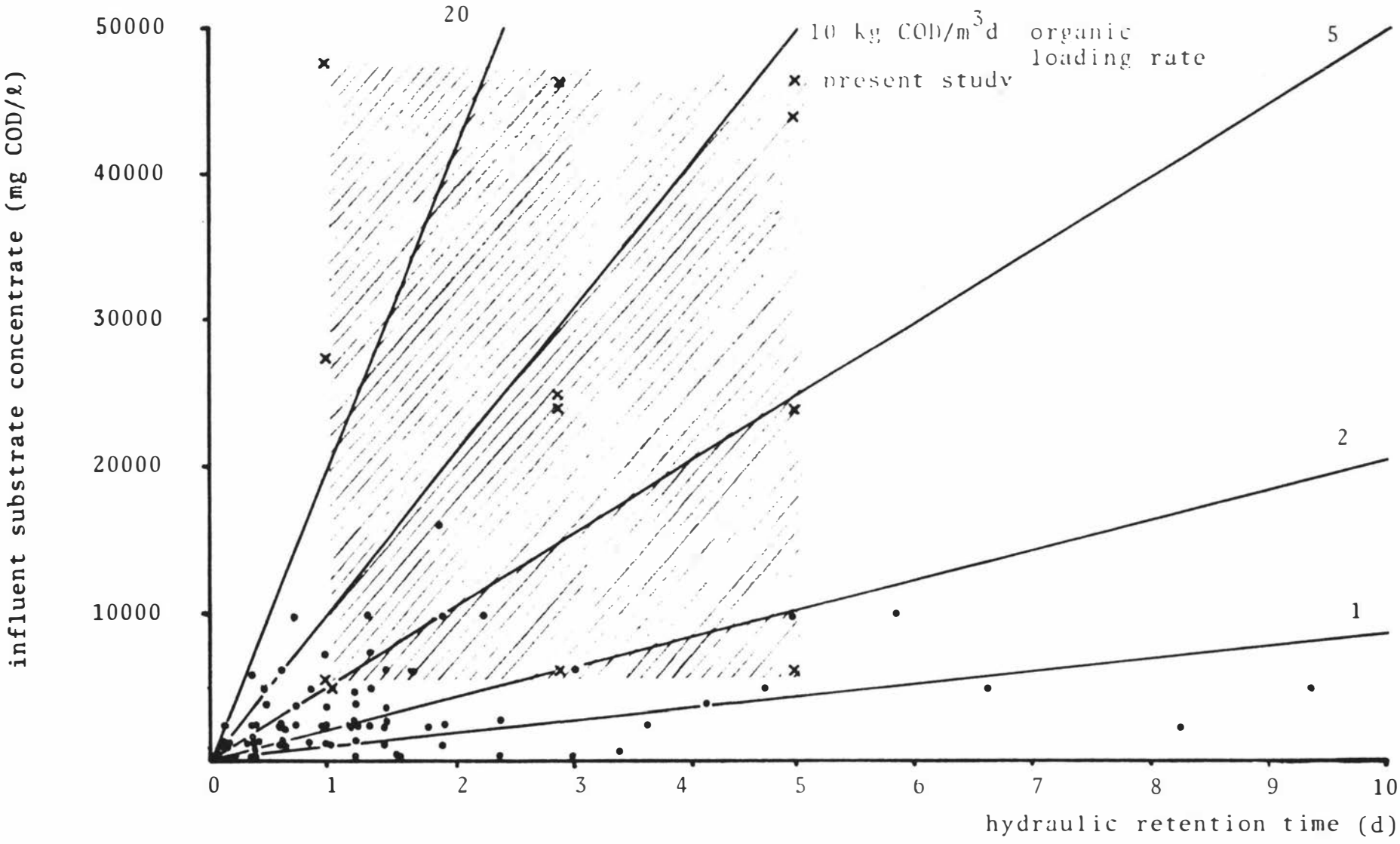


FIGURE 6.1: Scope of present study in the context of previous workers

streams such as plant wash waters. The previous studies may therefore be seen to be well extended by the present work.

6.2.1 Removal of Organic Material by the Anaerobic Filters

The removal of organic material as measured by the soluble COD of the filter feed and effluent concentrations has been presented in terms of residual (effluent) COD, percentage COD removal and rate of COD removal, and all three as functions of the influent substrate concentration, hydraulic retention time and combined organic loading rate (Figures 5.1 to 5.9).

Effluent COD Concentration

Figures 5.1 to 5.3 show that the amount of organic material in the filter effluent stream was almost completely dependent on the influent substrate concentration. At two of the three levels of feed strength there was improvement in effluent quality when the retention time was extended beyond one day (Figures 5.2, 5.3). Actual improvements were: from 31090 mg COD/ℓ down to 26592 mg COD/ℓ (14.5 percent) at 9000 mg BOD_5/ℓ nominal feed concentration; from 17378 mg COD/ℓ down to 8978 mg COD/ℓ (48.3 percent) at 5000 mg BOD_5/ℓ; and 2020 mg COD/ℓ to 2825 mg COD/ℓ an apparent 40 percent decline in effluent quality at the 1000 mg BOD_5/ℓ level. All these changes occurred over a five-fold increase in hydraulic retention time.

There is not an inconsiderable degree of scatter in the data, however the curves drawn on the figure represent predicted performance based on a least squares analysis of the effluent COD data set as a whole and using the four predictors indicated in the appendix (Table A1). As a whole, the regression lines account for 99 percent of the variation (r^2 = .981). The largest residuals occur for runs 5 and 9 where the actual performances were respectively considerably poorer and better than predicted. Table A1 also quantifies the relative significance of the influent substrate concentration (t ratio significant at >97.5 percent).

The extreme dependance of effluent COD on feed concentration is however a function of the wide range of conditions chosen for the trials rather than the operation of the filters themselves.

In previous studies there is some difference in relationship between the effects of the input parameters on effluent COD. For a pharmaceutical waste over the range 1000 to 8000 mg COD/ℓ similar results to the present study were noted (Jennett & Dennis, 1975), for a synthetic waste, a linear relationship was drawn between influent and effluent COD at influent COD up to 10000 mg/ℓ (El-Shafie & Bloodgood, 1973) compared to the predicted curvature in Figure 5.2. No improvement at all in effluent quality was noted for a milk waste (Peterson, 1975) with a four fold increase in retention time from 8 to 30 hours at constant organic loading rates of 1.75 and 3.50 kg/m^3d. In this last study, remarkably constant effluent COD's were apparent as the influent COD and retention time were simultaneously manipulated to maintain a constant loading rate.

Similar manipulation in the present study (runs 1, 2 and 8, Table 5.1) indicated no such constancy, a phenomenon also noted in two other studies (Young & McCarty, 1967) and Jennett & Dennis, 1975). Therefore the use of the common waste treatment parameter, organic loading rate, is not an appropriate predictor for effluent substrate concentration.

Figures 5.2 and 5.3 imply that there was a readily degradable portion present in the waste stream which was metabolised in about one day after which more resistant compounds were attacked. This concept will be further discussed in a following section.

The importance of effluent COD lies in its effect on the receiving points downstream of the anaerobic filter, such as the effects of shock loading a receiving water or

downstream biological treatment in the case of discharge to a municipal sewer. However the benefits obtainable by extending the detention time beyond one day would surely be offset by the increased cost of reactor size. Economics of reactor size are dependent on the amount of material removed and the rate at which this occurs.

Percentage Removal of COD

Figures 5.4 to 5.6 describe the effects of the primary variables on percentage removal of COD by the anaerobic filters. Figure 5.4 indicates a trend towards reduced fractional removal at higher loading rates and Figures 5.5 and 5.6 show that increasing the feed concentration up to 9000 mg BOD_5/ℓ and reducing the retention time from 5 to 1 day both exert a negative effect on the resulting fraction of material removed.

Although carried out at lower influent concentrations (and often shorter retention times) than in the present study this negative relationship between fractional removal and loading rate has been noted by all previous studies (Chapter 2). A linear decrease in one case (Young & McCarty, 1967) and an implied linearity in others at constant influent substrate concentration has led to the application of first order kinetic models for the anaerobic filter. An attempt has been made to define the kinetic constants for previous studies in chapter 2 of this work.

The data from this study do not show a good agreement between observed and predicted performance. This is due to the wide scattering evident in the relevant figures. Table A2 shows that the least squares estimates on four predictors, including one for the different filters used, covers only 84 percent of the observed variation (r^2 = .716). Run 3 had a large difference between observed and predicted values. Of the three levels of influent substrate concentration used the results at the highest, 9000 mg BOD_5/ℓ are the most consistent.

At a constant loading rate of 1.0 kg BOD_5/m^3d there was good agreement between the runs 1, 2 and 8 with values of 60.8, 65.6 and 63.0 percent COD removed respectively.

Overall, the data appeared to contradict the observed removals at the lower feed concentrations previously tested, that is either independence of percent COD removal with respect to loading rate at low loading rates (Young & McCarty, 1967) (2.38 to 4.76 kg COD/m^3d) and higher (Witt *et al*, 1979) (8-24 kg COD/m^3d); but percentage removal being better at high feed concentration/long retention time combinations at higher loading rates (Young & McCarty, 1967; Jennett & Dennis, 1975; Peterson, 1975; Johansen & Carlson, 1976). Similar trends to the present study have also been noted (Arora *et al*, 1975; Hudson *et al*, 1978). The latter of these noted large drops in COD removal of 80 to 52 percent at loadings of 0.15 to 0.36 kg COD/m^3d for each of two filters one packed with rock and the other with oyster shell and treating shellfish processing waste - a very poor performance compared to the removals at much higher rates in the present study.

In considering the relative magnitudes of percent COD removal, the type of waste being treated must be borne in mind and this is often the determining factor (Witt *et al*, 1979; Chian & de Walle, 1977). The relative BOD/COD ratios vary widely, for instance 0.52 for vegetable tannery waste (Arora *et al*, 1975), 0.9 for shell fish waste and approximately 0.2 for this study. The facility of determining COD rather than BOD_5 has probably favoured the use of the former in most cases.

Higher loading rate studies have found better COD removals than this study for instance 88 percent for piggery waste at 19.6 kg COD/m^3d (Colleran *et al*, 1982) and 46-60 percent for a synthetic waste (Mueller and Mancini, 1975), both of these materials were relatively readily degradable.

Rate of COD Removal

Given an amount of oxygen demand to be removed from a waste water stream, the rate at which it can be so removed is all important from an economic point of view, since the faster the removal rate the smaller the plant need be.

The relationship between applied and removed COD (Figure 5.7) implies a nearly linear increase. This is however erroneous as the slope at each point must equal the fraction COD removal which has been shown to vary widely over the range of conditions tested in this study.

There remains, nevertheless, the very strong correlation ($r = 0.989$) shown in Figure 5.7. It implies that the more highly one loads the system the faster it will react to remove the applied load. Interestingly, the different conditions tested which gave rise to the constant loading rate conditions (1.0 kg BOD_5/m^3d) showed agreement in removal rates to within six percent. The increased removal rate achieved by reducing the hydraulic retention time to 1 d compared to using 3 or 5 d is apparent in Figure 5.8 and the effects of both hydraulic retention time and influent substrate concentration are greatest at their respective levels which give rise to the highest organic loading rate.

When the data from Table 2.6 is manipulated to generate COD removal rates and those rates are plotted against applied loading rate (Figure 6.2), although there is considerable spread in the plot the generally increased removal rate with higher loading rate is immediately apparent. The spread in data points is to be expected due to the large number of different filter sizes, packings, loadings and feed materials as used by the different workers. Young & McCarty (1967)'s data is plotted as groups A & B and lines drawn in by eye indicate a slight concavity towards the abscissa for the protein/carbohydrate waste (A) but not so much for the volatile

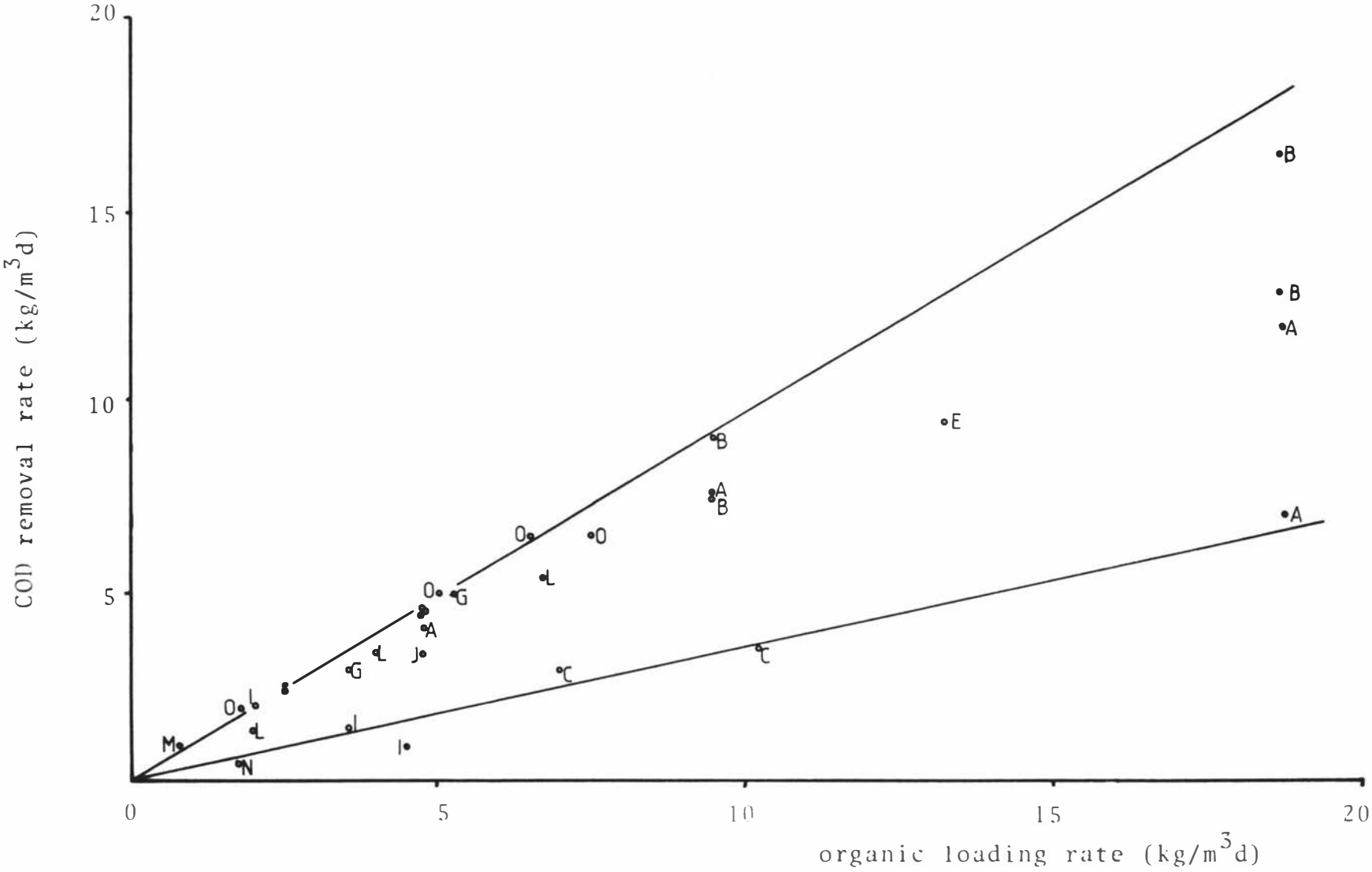


FIGURE 6.2: Relationship between applied COD and COD removal rate for data in Table 2.6

acid waste (B). The data from this present study which is also a carbohydrate waste also shows a concavity towards the abscissa and therefore the process must be reaching a limit in respect of treatment capacity at the highest loading in each case. An operating envelope drawn on Figure 6.2 shows that over the range of loading rates up to 20 kg COD/m^3d, expected removals of COD fall in the range 35 to 95 percent. The present study is seen to extend the upper range of Figure 6.2 but along the lower limit of the operating envelope proposed (Figure 6.3). This would be due to the relatively low degradability of the yeast waste since those wastes represented near the upper bound are the volatile fatty acid waste of Young & McCarty (1967) and the synthetic waste of El Shafie & Bloodgood (1975) at the higher loading rates. In the present study the decreasing removal rate occurred at loadings above about 4 kg BOD_5/m^3d (which corresponded to approximately 20 kg COD/m^3d) compared to about 9 kg COD/m^3d for the protein carbohydrate waste. The volatile acid waste (B in Figure 6.2) shows little decrease in removal at loadings up to 18.75 kg COD/m^3d and therefore the system was more amenable to this type of waste than the protein/carbohydrate one.

Other salient features of Figure 6.2 are the rapid decrease in rate of removal with increased loading for the carbohydrate waste treated by Plummer *et al* (1975) (Plot C) at a very low loading rate. Similar trends to the one found in this study were also observed by Jennett & Dennis (1975), Plot D; Peterson (1975), Plot G; Hovious *et al* (1971); van Velsen *et al* (1979) Plot L; Norman & Frostell (1977) Plot M.

The vegetable tannery liquor treated by Arora *et al* (1975), Plot I, shows a peak removal rate at 2.1 kg COD/m^3d loading rate and a decline after that possibly due to toxicity influences of the tanning liquor.

From a kinetic viewpoint it is important to know the relationship between loading rate and removal rate so

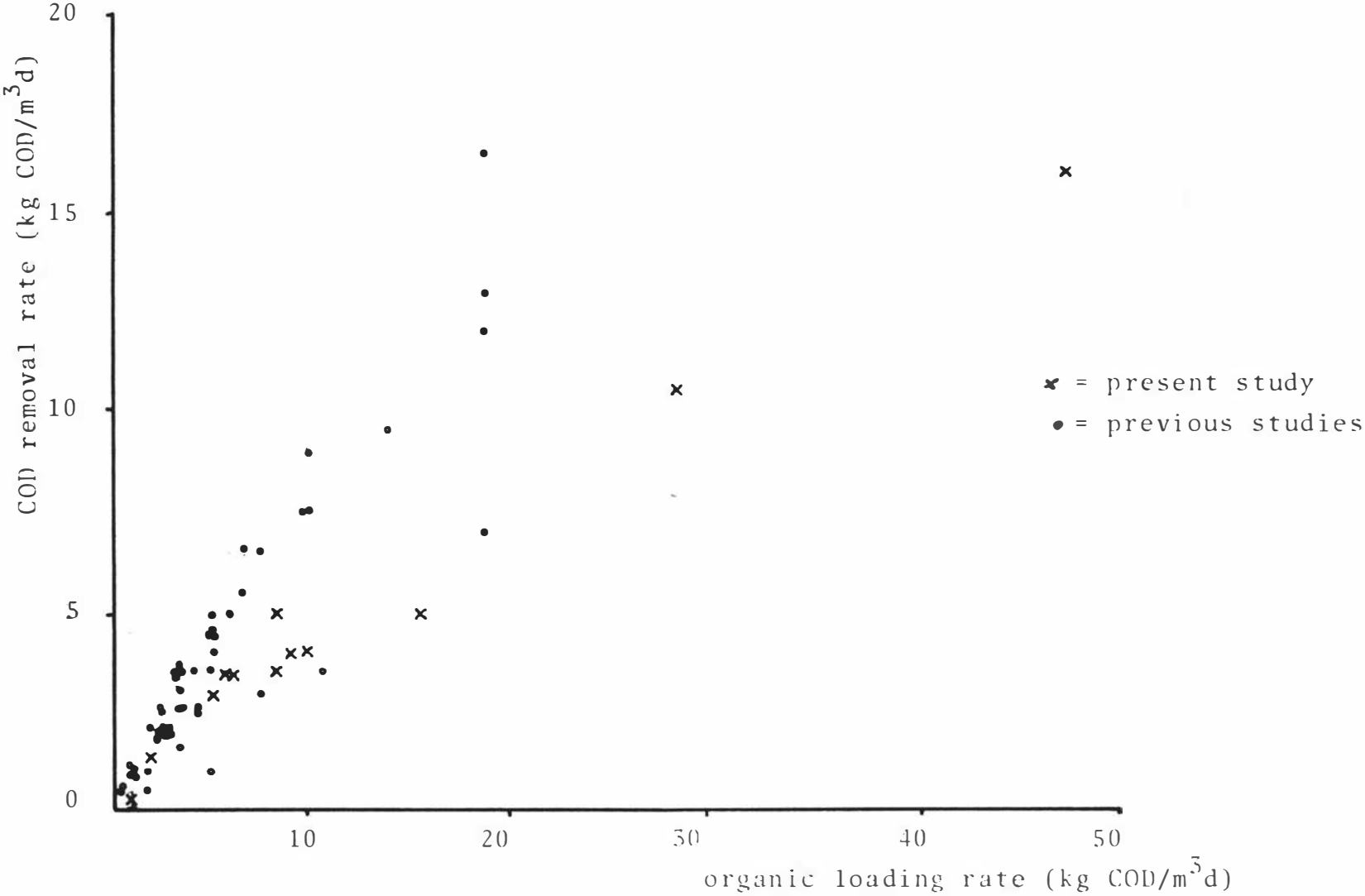


FIGURE 6.3: Effect of COD loading rate on COD removal rate for data in Table 2.6 and present study

that an optimum size processing facility can be designed.

The use of the COD removal rate data to provide a model for use in prediction of that rate will be discussed in a later section.

6.2.2 Conversion of Organic Material to Methane

The conversion of organic material to methane is a complex and little understood process especially with respect to complex waste water streams. The scope of this study does not extend to discussion of the microbiological or biochemical aspects of methanogenisis. However, the practical aspects are vital. Soluble organic material removed from the waste stream is utilised either for cellular growth or converted to methane. Since methane is a high energy compound, little energy is normally available for cell growth in a balanced anaerobic digestion process. Production of biomass has been shown to be dependent on the type of compound being catabolised (Speece & McCarty, 1962). The solids retention time also has a direct influence. Digester studies on compounds as carbon and energy substrates indicated a net synthesis from substrate COD of 4.5 to 6 percent for fatty acids and glycine; and 26 to 42 percent for glucose starch and cellulose and all at 5 d solids retention time (SRT). When the SRT was extended to 30 d, the solids synthesis dropped in both cases to 3 percent for the fatty acid material and 10 to 17 percent for the carbohydrate. In the anaerobic filter treating yeast waste, which is high in carbohydrate, a considerable amount of the removed COD may be expected to appear as net solids synthesis, even at the high SRT found in the process.

From a mechanical viewpoint it is essential that solids production be kept to a minimum to maintain the void volume in the filter and reduce the requirement for solids wasting.

The anaerobic filter does not lend itself well to solids analysis during operation. A solids balance was made by Young & McCarty (1967) in their first study by dismantling the filter after the runs and measuring the solids accumulation. That practice was not considered to be feasible in the present study due to the attendant time requirement for reseeding and re-establishment of an active biomass. An indirect measurement of solids systhesis has been used in several studies (Peterson, 1975; Mosey, 1977; Frostell, 1981) whereby the observed ratio of conversion of COD to methane is compared with a theoretical maximum, usually 0.351 m^3 methane/kg COD removed (McCarty, 1964). A ratio near unity is taken to mean little net solids synthesis has occurred.

The fractional conversion of COD to methane (FTCM) cannot be calculated without detailed data on gas production and methane content, which has been generally lacking in the literature to date. The reason is probably the consideration of the gas as merely a useful by-product of waste treatment. In the present context of energy awareness, generation of methane is considered a valuable resource (Trevelyan, 1975; Frostell, 1979; Colleran, 1982) and in the present study, equal emphasis was placed on the gas production as COD removal to assess the anaerobic filter's efficiency as an energy recovery process. The FTCM is therefore of considerable importance.

While it is apparent from the results that the maximum productivity of total gas and methane occurred at the highest loading conditions tested, that is 1.552 ℓ/ℓd of methane at 9.0 kg BOD_5/m^3d (47.2 kg COD/m^3d), this was only marginally better than gas productions as observed between 2 and 4 kg BOD_5/m^3d (Figure 5.20). Further, the much higher COD removal rates at the highest organic loading rate (Figure 5.7) indicate that the trend towards lower conversion efficiency of COD to methane as loading rate increased noted in Chapter 2 (Table 2.9) is even more marked at loading rates beyond those previously examined.

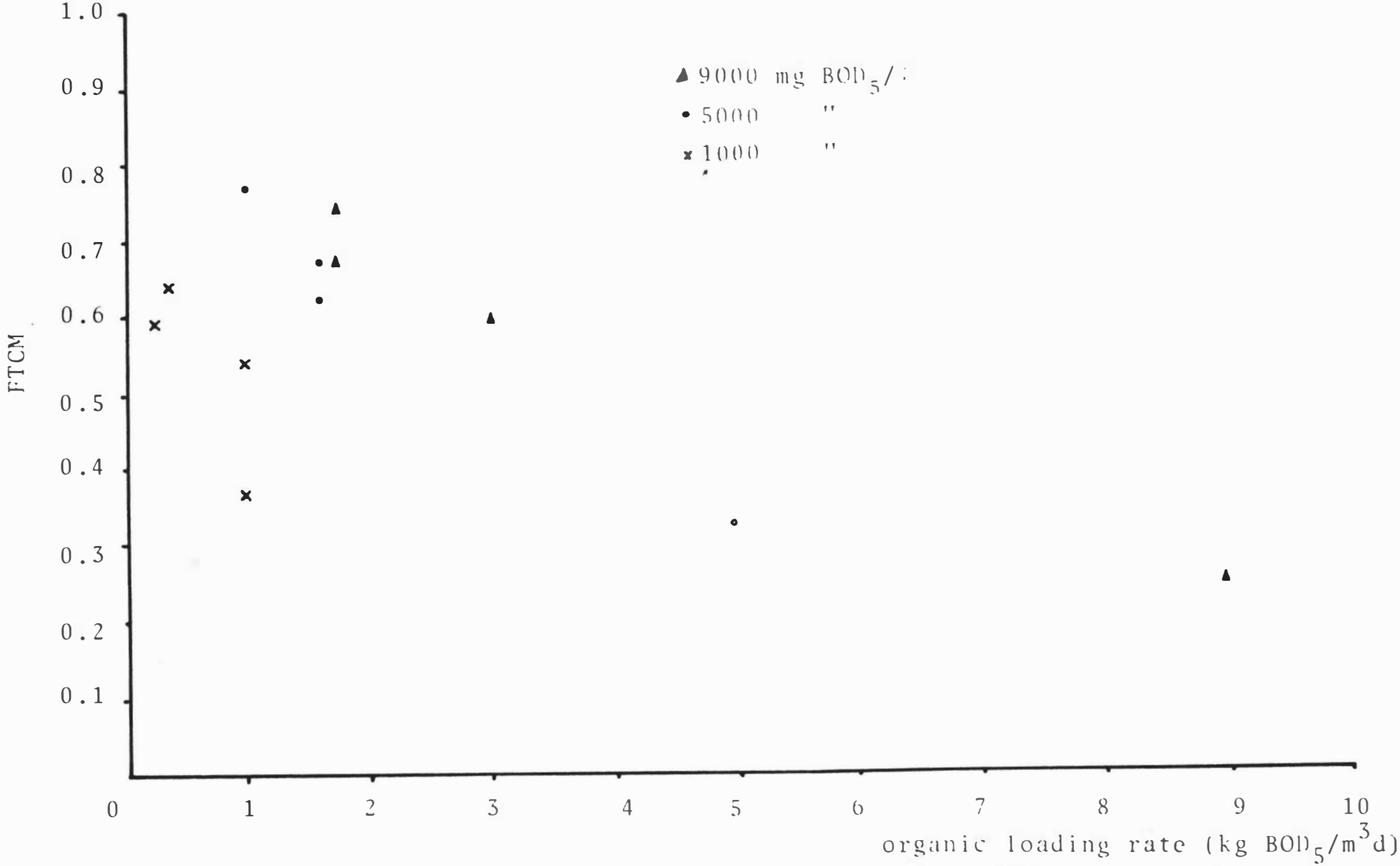


FIGURE 6.4: Fraction of theoretical conversion of COD to methane (FTCM) as a function of organic loading rate

From Figure 6.4 it could be deduced that, at loading rates above about 10 kg BOD_5/m^3d, the anaerobic filter is operating as a sludge generator, and that the non-methanogenic bacteria are prevailing in an unbalanced digester.

With a conversion ratio of 24.6 percent of the theoretical amount (run 9), 75.4 percent of the COD removed is unaccounted for in the methane, and discounting the possible losses of gas in the system, is probably present as accumulated solids in the filter. The best conversion efficiency occurred in run 5 at 77.2 percent of the theoretical maximum. The magnitude of all the conversion rates is within the range of those previously reported for other waste types. A mathematical model to predict conversion rate is suggested in the following chapter. Some actual solids balances published have noted syntheses of 3 percent for pharmaceutical waste (Jennett & Dennis, 1975) and 17 percent for protein carbohydrate and 2 percent for volatile acid waste (Young & McCarty, 1967). These were all consistent with those noted by Speece & McCarty (1962).

Therefore, in considering the anaerobic filter for the treatment of high strength yeast plant effluent, although the highest organic removal rate and methane production rate occurred at a loading rate of 9.0 kg BOD_5/m^3d (47.2 kg COD/m^3d), better efficiencies of conversion were manifest at lower loading rates, especially those at longer than one day hydraulic retention time. To compete with sludge bed processes Frostell (1981) concluded that the anaerobic filter must be able to operate at greater than 5-10 kg COD m^3d which the present study has shown to be possible.

6.3 Environmental Factors Within the Anaerobic Filter Process

6.3.1 Volatile Acid Content

The importance of volatile acidity in anaerobic digesters is apparent both from the point of view of pH and

toxicity of the acid themselves.

The magnitude of the volatile acid concentration would not necessarily be considered inhibiting even at the 4846 mg/ℓ (as acetic acid) observed in the trial at 9 kg BOD_5/m^3d (47.2 kg COD/m^3d). Volatile acid concentrations of 8400 mg/ℓ have been reported as not impeding digestion (Augenstein *et al*, 1976/77) in the anaerobic treatment of solid wastes. In another study (Hobson & Shaw, 1976) the organism *Methanobacillus formicicum*, considered to be typical of anaerobic digestor biomass was found to be unaffected by acetic and butyric acid concentrations of up to 10,000 mg/ℓ. However, propionic acid extended the lag phase in batch culture from 4 to 6 days at concentrations of 1000 and 5000 mg/ℓ respectively. It is therefore possible that the propionic acid concentrations observed in this study were exerting a negative influence in the operation.

6.3.2 Ammonia

Ammonia is important from the nutrient point of view as a nitrogen source from the biomass and also ammonia exerts a toxic effect on the biomass at higher concentraions. The toxic concentrations have variously been noted as 1500 to 3000 mg/ℓ (McCarty, 1964) and 4000 mg/ℓ (Hobson & Shaw, 1976) whereas other work in piggery waste noted no inhibition by ammonia at 5 g/ℓ although an extended lag phase was noted. The inhibiting level of ammonia appeared to be largely associated with the acclimation level of the biomass (van Velsen *et al* 1979).

Ammonia also exerts a third influence in anaerobic digestion in that it provides the primary pH buffer

system (Gossett *et al*, 1978). In association with carbon dioxide (equation 6.1), 1g of ammonia gives rise to 3.6g of bicarbonate alkalinity

$$NH_3 + CO_2 + H_2O \rightleftharpoons HCO_3^- + NH_4^+ \qquad (6.1)$$

Melleringer & Donellor (1971) found that ammonia nitrogen gave rise to 23 percent of the total alkalinity of anaerobic digestion. Kroeker *et al* (1979) aver that the mechanisms of process stability are bio-chemically related to the concentration of ammonia in solution.

In the present study, the principal concern with respect to nitrogen in the feed material was that there be sufficient without augmentation to maintain nutrient levels for the biomass. The ratio of COD:N in the feed material was 100:2 and the presence of ammonia in the filter environment (Figure 5.29) was taken to indicate that there was no lack of nutrient nitrogen.

The applied COD:N ratio was not as low as one previous study 100:0.37(de Walle & Chian, 1976) which concluded there was no nutrient lack. None of the ammonia concentrations measured in this present study were near those considered to be inhibitory, the highest being 18.1 mM (308 mg/ℓ)

6.3.3 The Effects of Sulphate in the Feed

The high concentrations of inorganic sulphates encountered in molasses based wastes such as yeast plant effluent and distillery slops have long been known to cause problems in anaerobic digestion (Sheehan & Greenfield, 1980; Basu & Leclerc, 1972; Stander *et al*, 1950)

The sulphate reducing bacteria found in the digestor biomass readily reduce the sulphates to sulphides, which are toxic and also the sulphate reducing reaction may compete with methanogensis for available hydrogen thus reducing methane yields.

In the present study, the total suphide concentrations in the anaerobic filters were largely correlated with influent substrate concentration and, therefore sulphate concentration in the feed. The influent COD-sulphate correlation was r=0.99 and sulphate-sulphide r=0.706. From Figure 5.30 the effect of influent sulphate concentration is noted. The sulphide concentration of 186 mg/ℓ at 9 kg BOD_5/m^3d, lies within the somewhat broad range of sulphide concentrations of other runs at the same applied concentration even though the total amount of sulphate loaded on the system was some five fold higher.

There is some controversy over the concentrations at which sulphides be toxic or inhibitory to anaerobic digestion. Early reported values of 150 mg/ℓ (McCarty, 1964; Stander *et al*, 1950) were found to be inhibitory, but low concentrations of 5 mg/ℓ to stimulate digestion (Winfrey & Zeikus, 1979). Studies on anaerobic digestion of yeast plant effluent (Stander *et al*, 1971) found that additions of sulphate of up to 18000 mg/ℓ which gave rise to 400 mg $S^=/ℓ$ in the digestor did not appear to cause inhibitions. Batch studies using *Methanococcus* spp on media containing 1g $Na_2S/ℓ$ demonstrated further tolerance (Abram & Nedwell, 1978), *Methanobacillus* MOH has been shown to be tolerant of H_2S at (680mg/ℓ) and pH 6.6/6.8.

Sulphate is thought to compete for hydrogen in the anaerobic environment. In fact a waste treatment process was tested in which sulphate was added to a waste stream and sulphate reducing bacteria utilised organic material and reduced sulphate to hydrogen sulphide. At 2.56 kg VS/m^3d, 32 percent of the COD of the waste was removed and about 400 mg/ℓ.d of sulphide produced (Pipes, 1960). Several relationships have been proposed for the effects of sulphate on methanogensis such as a commensalism between *Desulphovibrio* spp. *Methanobacillus* spp. due to acetate production by the former and use by the latter, but with inhibition of the methanogen in the presence of sulphide (Cappenberg, 1975). However, in the presence of sulphate, the lack of

methane production in previously synergistic relationship between *D. velgaris* and a bacteria similar to *M. formicium*, was due to competition for H_2 rather than sulphate or sulphite toxicity (Bryant *et al*, 1977; Winfrey & Zeikus, 1979). Methane production may also be reduced due to competition for acetate as well as hydrogen, as the free energy change for reduction of sulphate by acetate is more favourable than acetate reduction (Equations 6.2 and 6.3)(Kahan & Trottier, 1978),

$$SO_4^{=} + CH_3COO^- \quad HS^- + 2HCO_3^- + \Delta G_o' = -47 \text{ kJ} \qquad (6.2)$$

$$CH_3COO^- + H_2O \quad CH_4 + HCO_3^- \quad \Delta G_o' = -31 \text{ kJ} \qquad (6.3)$$

However, an accumulation of volatile acids has also been shown to inhibit sulphide production (Basu & Ghose, 1961),

From a practical point of view it is advisable to reduce sulphide concentrations due to their malodourous and toxic nature (Stander & Gien, 1956) and deleterious effects on concrete and other construction materials (Ruffer, 1978). Removal of sulphide is generally achieved by precipitation with heavy metal ions. In fact, toxicity due to either species is treated by addition of the other (Lawrence & McCarty, 1965; Masselli *et al* 1967)

The benefits of reducing the sulphide concentration of the present study, and thereby to ascertain whether sulphide was exerting a toxic effect on the filter operation, were assessed. The addition of ferric chloride at 2.5 g/ℓ (equivalent to 7 mmol Fe/ℓ) effectively reduced the filter sulphide concentration to less than 5 mg/ℓ. There was, however, no improvement in filter performance associated with the low sulphide concentration and this was taken to indicate that sulphide toxicity was not a problem.

Hydrogen sulphide was not detected in the filter off-gases at any time during the operation, but must have been present. Typical H_2S contents have been noted as less than 0.5 percent (v/v) for molasses based fermentation wastes (Stander *et al*, 1950) and less than one percent for yeast plant effluent (Stander *et al*, 1971). The absence of H_2S from the digester gas is desirable to prevent corrosive effects of sulphur oxides when the gas is burnt for heating purposes.

The hydrogen sulphide may be stripped from the gas, however this is expensive. Alternatively a "stable anaerobic microbial association" has been developed (Anderson *et al*, 1982) which maximises the production of methane in the presence of sulphate in molasses based wastes. The process claims no free H_2S detectable.

No stimulation of digestion was noted after the additions of iron as was noted in previous studies where stimulus of acetate conversion aided in reduction of volatile acid concentration (Speece & McCarty, 1962; Hobson & van den Berg, 1979). However, there would have been only a slight excess of Fe(II) ions assuming a 200 mg/ℓ sulphide concentration to be removed.

6.4 The Anaerobic Filter as a Pre-treatment for Yeast Plant Effluent

The operating conditions for the anaerobic treatment of yeast plant and similar waste waters is illustrated in Figure 6.5. Although the BOD_5 values for other studies have been much higher than those in the present study, the extended retention times used (up to 25 d) meant that no loading rates have exceeded 4 kg BOD_5/m^3d. This is probably due to the inability of stirred tank anaerobic digestors to maintain biomass at the high hydraulic loadings which accompany high organic loading rates.

The longer hydraulic retention times have, however, contributed to very high percentage removals of BOD_5 ,

removals in excess of 90 percent at retention times greater than 10 days.

Because of the low removal efficiencies obtained in the anaerobic filter, the process design could not be considered for more than an on site pre-treatment plant. However, at the high loading rates permissible on the filter a small treatment unit would be able to pre-treat a large amount of effluent in a relatively short time. For instance a previously predicted maximum organic loading rate for molasses waste was 3.2 kg BOD_5/m^3d at which BOD_5 removal was 74 percent however the methane content of the gas was 30 percent and conversion only .077 m^3CH_4/kg BOD_5 removed (Basu & LeClerc, 1972). On the anaerobic filter a similar conversion rate was found but at a loading rate of 9 kg BOD_5/m^3d, corresponding gas content was 50 percent methane. COD removals were similar on the anaerobic filter to the stirred tank reactor for yeast plant effluent (Hansford & Richtor, 1975) but at greater loading rates viz 67 to 71 percent COD removal at loading rates of 2.14 to 11.35 kg COD/m^3d on the stirred tank and 34 to 67 percent removal on the filter at loading rates of 1.15 to 47.2 kg COD/m^3d.

As well as not requiring the size of conventional anaerobic digestors, the anaerobic filters would not require the technical and mechanical sophistication of some of the other waste treatment systems such as the 'ANAMET' process (Huss, 1977; Skogman, 1979). Also, since yeast waste contains nothing of value recoverable for animal feeds (Jackson & Lines, 1970) its only other value is as water for paddock irrigation, a disposal method of limited effectiveness in winter months on already saturated ground.

CHAPTER SEVEN

MODELLING OF THE ANAEROBIC FILTRATION OF YEAST WASTE

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MODELLING OF THE ANAEROBIC FILTRATION OF YEAST WASTE

7.1 Introduction

The steady state data gathered in the runs involved in the experimental design were used to produce response equations for the process of anaerobic filtration of yeast plant effluent. There were two reasons for these models. Firstly to produce statistically based mathematical models which would describe the observed process. Although a number of anaerobic filter studies have been made to date, there is no simple quantitative basis which can be used for design purposes. Secondly, a quantitative model was desired which could be used to predict the optimum operating conditions within the experimental range for two runs subsequent to those of the experimental design.

Four system response parameters which are of engineering design significance were chosen to be modelled. These were rate of removal of COD, total gas production, methane production and conversion of COD to methane expressed as a fraction of the theoretical maximum conversion obtainable.

Models previously applied to the anaerobic filter, and other waste treatment systems have usually been based on kinetic models of chemical reactions or bacterial growth and are often extremely complex and of little use for practical design problems (Mueller & Mancini, 1975). The statistical basis of the experimental design used in the present study was chosen for simplicity and to enable the results to be easily utilised in the fitting of regression models.

The models presented in this chapter are the results of polynominal regression of the data. The analysis was carried out using the 'MINITAB' statistical package

TABLE 7.1: Response Data and Coded Experimental Design Factors Used in Regression Analyses

Run	ISC	HRT	TEMP	AF	COD rem. kg/m^3d	GAS ℓ/ℓd	METHANE ℓ/ℓd	FTCM
1	-1	-1	-1	-1	3.336	0.845	0.706	0.541
2	-1	-1	-1	1	3.599	0.599	0.519	0.369
3	-1	0	1	1	1.320	0.409	0.335	0.651
4	-1	1	1	-1	0.590	0.173	0.138	0.600
5	0	-1	1	1	10.427	1.770	1.343	0.331
6	0	0	-1	-1	3.557	1.296	0.859	0.618
7	0	0	1	1	5.019	2.010	1.337	0.682
8	0	1	-1	-1	3.054	1.350	0.917	0.772
9	1	-1	1	-1	16.08	3.110	1.552	0.246
10	1	0	-1	-1	5.204	2.153	1.214	0.597
11	1	1	-1	1	3.754	1.640	1.105	0.756
12	1	1	1	1	3.767	1.784	1.309	0.887

release 81.1 Pennsylvania State University, (Ryan *et al*, 1976) operating on the Burroughs B6700 computer at Massey University. The response equations represent parsimonious models which best fit the data. All regressions were carried out using the coded levels of the dependant variables this giving equal weighting to each of those variables. The coded models were then decoded using the equations described in Chapter 4.

The criteria used to assess the fit of a model were the statistical significance of the predicted coefficients, and the coefficient of determination (r^2). This latter is the ratio of the variance explained by the regression equation to the total variance, viz

$$r^2 = \frac{ss_{regression}}{ss_{total}} \qquad (7.1)$$

and is the square of the correlation coefficient between the observed and predicted values of the response variable.

The data used for the regression models are presented in Table 7.1 and the regression parameters for the models in Tables 7.2 to 7.5

7.2 The System Response Equations

7.2.1 COD Removal Rate COD(r)

The parsimonious model for COD removal rate is expressed as kg COD removal/m^3 filter void volume per day in its coded form is

$$COD(r) = 5.381 + 3.405isc - 3.637hrt + 0.803hrt^2 - 2.432(isc)(hrt) \qquad (7.2)$$

Upon decoding the equation 7.2 becomes

$$COD(r) = 5.837 + 1.764ISC - 3.914HRT + 0.603HRT^2 - 0.304(ISC)(HRT) \qquad (7.3)$$

where isc = coded level of influent substrate concentration

ISC = influent substrate concentration in g BOD/ℓ (1 < ISC < 9)

hrt = coded level of hydraulic retention time

HRT = hydraulic retention time in days (1 < HRT < 5)

Regression parameters for this model are presented in Table 7.2.

From Table 7.2 it is apparent that the four factors used to predict COD removal rate have very sound statistical basis, with confidence levels 99.9 percent for all except $(HRT)^2$ for which the level is 99 percent. The equation explains greater than 95 percent of the variation observed in the data and the 95 percent confidence interval applicable to that data is $\pm$ 2.31 kg COD (r)/m^3d.

COD removal rate is therefore seen to be a function of substrate concentration, HRT, an interaction between these two and the curvature in the data attributed to a quadratic term in HRT.

7.2.2 Total Gas Production

The total gas production in volumes of gas per unit net volume of filter per day in coded form is defined by

$$GAS = 1.428 + 0.934isc - 0.406hrt + 0.114temp - 0.089isc^2 \quad (7.4)$$

Upon decoding the gas production was

$$GAS = -0.858 + 0.401ISC - 0.203HRT + 0.046TEMP - 0.017ISC^2 \quad (7.5)$$

The variables are the same as for equations 7.2 and 7.3 and in addition, temp and TEMP are the coded and uncoded forms of the temperature factor, where 30 < TEMP < 35. The relevant regression parameters are presented in Table 7.3 from which it is seen that there is good evidence

for inclusion of the constant, ISC and HRT coefficients in the equation but only 90% confidence in the temperature and quadratic terms. The reason for retaining these latter two terms in the model is that it improves the coefficient of determination to 93.2 percent from less than 90 percent. The relatively small magnitude of the coefficients for these parameters mean they have little influence on the response and the equation is essentially linear. The 95 percent confidence interval for GAS is $\pm$ 0.556 ℓ/ℓd.

7.2.3 Methane Production

The parsimonious regression equation for methane production in its coded form is

$$METHANE = 0.945 + 0.486isc - 0.203hrt - 0.085isc^2 \quad (7.6)$$

Upon decoding the equation is

$$METHANE = 0.414 + 0.280ISC - 0.016ISC^2 - 0.101HRT \quad (7.7)$$

where the variables are the same as the previous models.

Regression data is presented in Table 7.4. All parameters in the response equation have a very good statistical basis for their inclusion with a minimum confidence level of 97.5 percent. The 95 percent confidence interval for methane production is $\pm$ 0.311 ℓ/ℓd and 91.6 percent of the observed variance is explained by the equation. Although the coefficient for the quadratic term is an order of magnitude smaller than the others its statistical basis is significant and hence the curvature in the data is assigned to the square of the substrate concentration.

7.2.4 Conversion of COD to Methane

For this model, the actual conversion of COD to methane is expressed as a fraction of the theoretical amount, based on

the stoichiometry of methane oxidation, determined by McCarty (1964) of 0.351 m^3/kg COD removed. The data could equally well have been left as the actual amount of methane produced per kg COD removed but it was felt that the factorial theoretical conversions conveyed more meaning about how effectively the process was operating.

The parsimonious regression equation for fractional conversion of COD to methane (FTCM) in its coded form is:

$$FTCM = 0.554 + 1.191hrt + 0.067temp - 0.064volume - 0.0416hrt^2 + 0.202(isc)(hrt) \quad (7.8)$$

where in addition to those variables already defined 'volume' is the coded level of filter volume.

The regression data is presented in Table 7.5. As well as a constant term, five parameters were found to be significant with minimum confidence level of 97.5 percent. The six term equation is able to account for 97.1 percent of the observed variation in the data and the 95 percent confidence interval on the predicted response is $\pm$ 0.089.

The large dependence of conversion rate HRT as noted in the graphical presentation of the data is quantified in Table 7.5, as is importance of the quadratic term hrt^2 with a 99.5 confidence level. There was a large interactive effect between HRT and substrate concentration.

It is apparent that some difference between the two filter units was manifest, although the coefficient is small relatively, statistical basis for its inclusion is sound (97.5 percent). The appearance of the term relating to particular filter means that one decoded response equation is not relevant. In the regression analysis this factor was arbitrarily coded using net filter volume as a basis although any classification could have been used as the factor is a qualitative one. Due to this lack of quantitative

TABLE 7.2: Regression Parameters for COD Removal Rate Model

Standard deviation of y about the regression line

s=1.13 with 7 degrees of freedom (d.f.)

Coefficient of determination r^2=0.955

Significance of the parameter estimates:

FACTOR	COEFFICIENT	95% CI $\pm$	t-RATIO	SIGNIFICANCE LEVEL
Constant	5.381	0.685	16.08	.001
isc	3.405	0.840	8.29	.001
hrt	-3.637	0.840	-8.85	.001
hrt^2	0.803	0.476	3.44	.01
isc x hrt	-2.432	0.982	-5.07	.001

Analysis of variance

DUE TO	D.F.	SUM OF SQUARES	MEAN SQUARE
regression	4	190.2	47.55
residual	7	8.87	1.27
total	11	199.07	

$t_{.001,7} = 4.785$ $t_{.01,7} = 2.998$

TABLE 7.3: Regression Parameters for Gas Production Model

Standard deviation of y about regression line
s=0.272 with 7 d.f.

Coefficient of determination r^2 = 0.932

Significance of parameter estimates:

FACTOR	COEFFICIENT	95% CI	t-RATIO	SIGNIFICANCE LEVEL
constant	1.4283	0.160	18.18	0.001
isc	0.9340	0.203	9.40	0.001
hrt	-0.4056	0.203	-4.08	0.005
temp	0.1144	0.160	1.46	0.1
isc^2	-0.0891	0.113	-1.60	0.1

Analysis of variance:

DUE TO	D.F.	SUM OF SQUARES	MEAN SQUARE
regression	4	7.128	1.782
residual	7	0.518	0.074
total	11	7.646	

$t_{.001,7} = 4.785$ $t_{.005,7} = 3.499$ $t_{.1,7} = 1.415$

TABLE 7.4: Regression Parameters for Methane Production Model

Standard of deviation of y about the regression line

$s = 0.152$ with 8 d.f.

Coefficient of determination $r^2 = 0.916$

Significance of parameter estimates :

FACTOR	COEFFICIENT	95% CI	t-RATIO	SIGNIFICANCE LEVEL
constant	0.945	0.090	21.57	.001
isc	0.486	0.203	8.77	.001
hrt	-0.203	0.203	-3.66	.005
isc^2	-0.085	0.063	-2.73	.025

Analysis of Variance :

DUE TO	D.F.	SUM OF SQUARES	MEAN SQUARE
regression	3	1.996	0.665
residual	8	0.184	0.023
total	11	2.180	

$t_{0.001,8} = 4.785$ $t_{0.005,8} = 3.355$ $t_{0.025,8} = 2.306$

TABLE 7.5: Regression Parameters for Conversion of COD to Methane Model

Standard deviation of y about the regression line

$s = 0.0438$ with 6 d.f.

Coefficient of determination $r^2 = 0.971$

Significance of parameter estimates:

FACTOR	COEFFICIENT	95% CI $\pm$	t-RATIO	SIGNIFICANCE LEVEL
constant	0.5540	0.028	39.62	0.001
hrt	0.1910	0.032	12.33	0.001
temp	0.0674	0.046	3.02	0.025
volume	-0.0645	0.046	2.89	0.025
hrt^2	-0.0416	0.019	4.41	0.005
isc x hrt	0.2017	0.073	5.64	0.001

Analysis of Variance:

DUE TO	D.F.	SUM OF SQUARES	MEAN SQUARE
regression	5	0.3871	0.0774
residual	6	0.0115	0.0019
total	11	0.3986	

$t_{.001,6} = 5.208$ $t_{.005,6} = 3.707$ $t_{.025,6} = 2.447$

continuity between the factor levels, two equations must be defined, one for each filter. The two equations will vary only in the constant term vis equations 7.9 and 7.10, the decoded response equations for the small and large anaerobic filters respectively.

$$FTCM(1) = -0.363 + 0.156HRT - 0.031HRT^2 + 0.025(HRT)(ISC) - 0.076ISC - 0.027TEMP \quad (7.9)$$

$$FTCM(2) = -0.492 + 0.156HRT - 0.031HRT^2 + 0.025(HRT)(ISC) - 0.076ISC - 0.027TEMP \quad (7.10)$$

The appearance of the ISC term in the decoded form of the equation but not in the coded form results from the algebra involved in decoding the HRT x ISC interaction.

7.3 Interpretation and Optimisation of the Models

Four models have been developed to explain the observed variation in the experimental results and these are capable of assigning over 90 percent of the variation to basically two input parameters, namely HRT and influent substrate concentration, with small influences by temperature and filter unit used in the cases of two of the models. It is therefore convenient to graphically present predicted system response in terms of those two prime inputs.

A computer program has been used to produce contour plots of predicted response at the different combinations of HRT and substrate concentration. In this program, the response is calculated using the two relevant parameters while other parameters, if present, are held at constant values. Therefore there are plots for both temperature levels in the GAS model and each combination of temperature and filter unit for the FTCM model. By following the contour any optimal conditions in the range of experimental factors can be seen. The program is written in FORTRAN 77 and was run on a PRIME 750 computer. By using these contour plots it is possible to illustrate independent effects of both HRT and substrate

concentration on the system without the need to use the often ambiguous term of organic loading rate.

Figure 7.1 is the plot for COD removal rate from which a local maximum was noted of 15.64 kg COD rem/m^3d at conditions of 1 d HRT and 9000 mg BOD_5/ℓ.

In this model, the effect of the quadratic term can be seen with the relatively flat area at low HRT and its steepening as HRT is reduced.

The gas production model is presented in Figures 7.2 and 7.3, being the response at 30 and 35^{o}C respectively. The local maximum occurs at the highest loading on the system of 1 d HRT and 9000 mg BOD_5/ℓ.

The methane production model is presented in Figure 7.5. The maximum operating conditions for this model, within the range tested is predicted as 1.537 ℓ/ℓd occurring at 1d HRT and 8800 mg BOD_5/ℓ. The similarity between the total gas and methane contour plots is evident. At higher substrate concentrations, the methane contour lines curve more about the maximum. This indicates the relatively smaller effect of HRT.

Figures 7.5 to 7.8 illustrate the conversion of COD to methane model at each of the temperature and filter unit combinations. The plots are identical in shape and vary only in the values on the contour lines due to changes in the equation's constant term.

The contour plots show a steeply dropping COD conversion efficiency as HRT is reduced while simultaneously increasing the substrate concentration. However, at the same high substrate concentration, by increasing retention time, the maximum conversion efficiency is predicted. A large flat area is predicted at the lower end of the concentration range and mid range HRT. This area constitutes one side

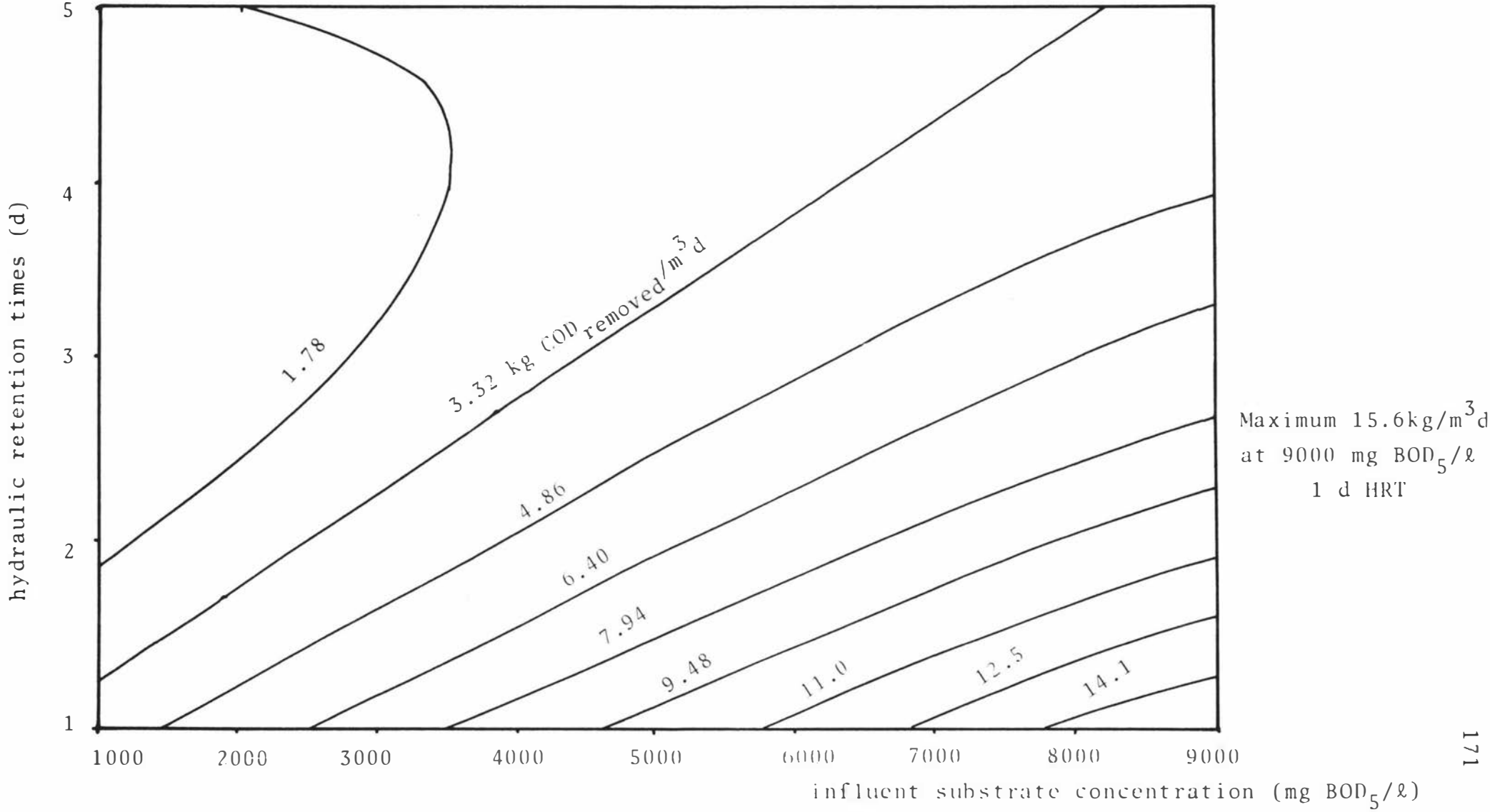


FIGURE 7.1: Contour of plot of COD removal model

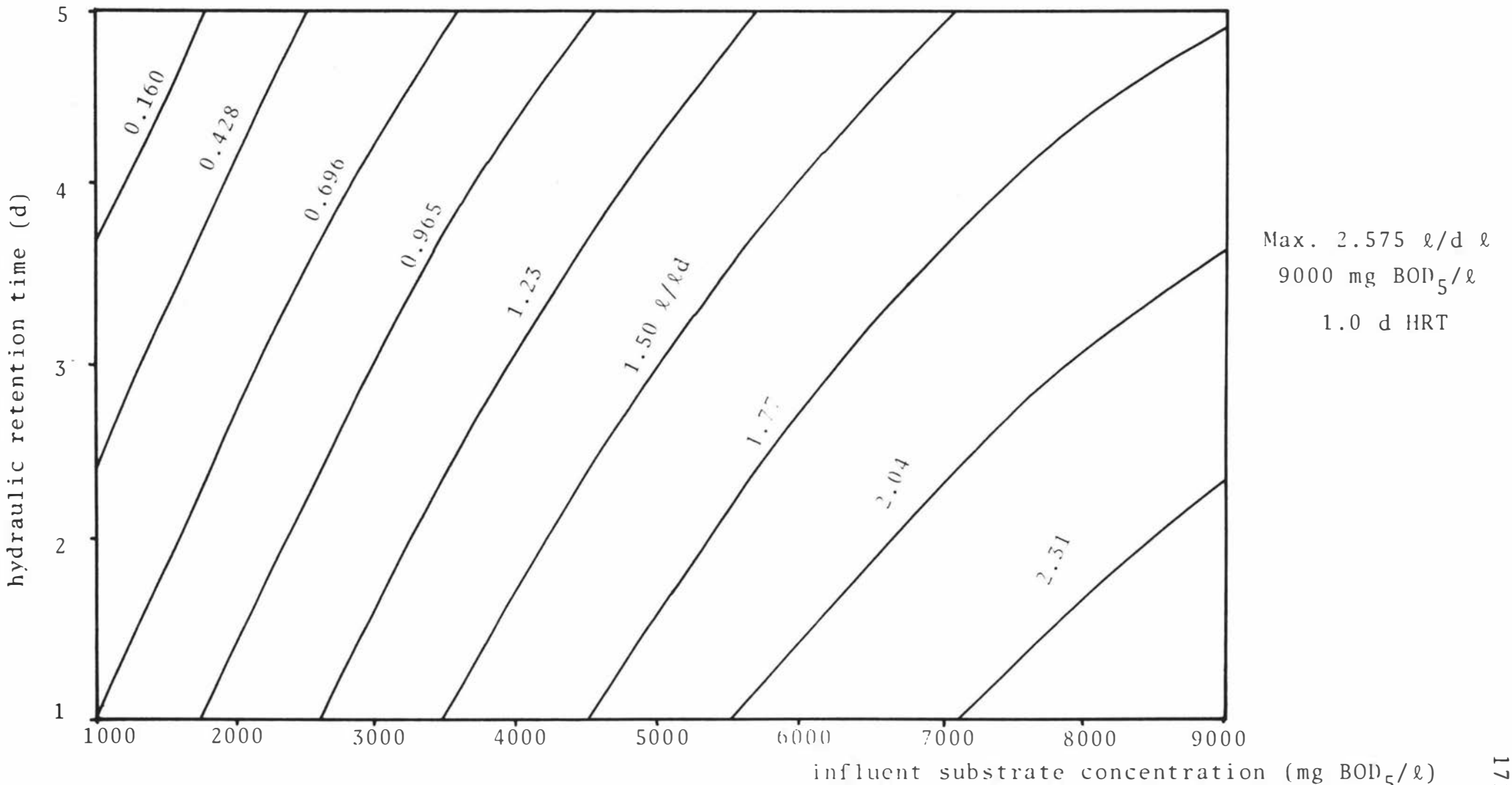
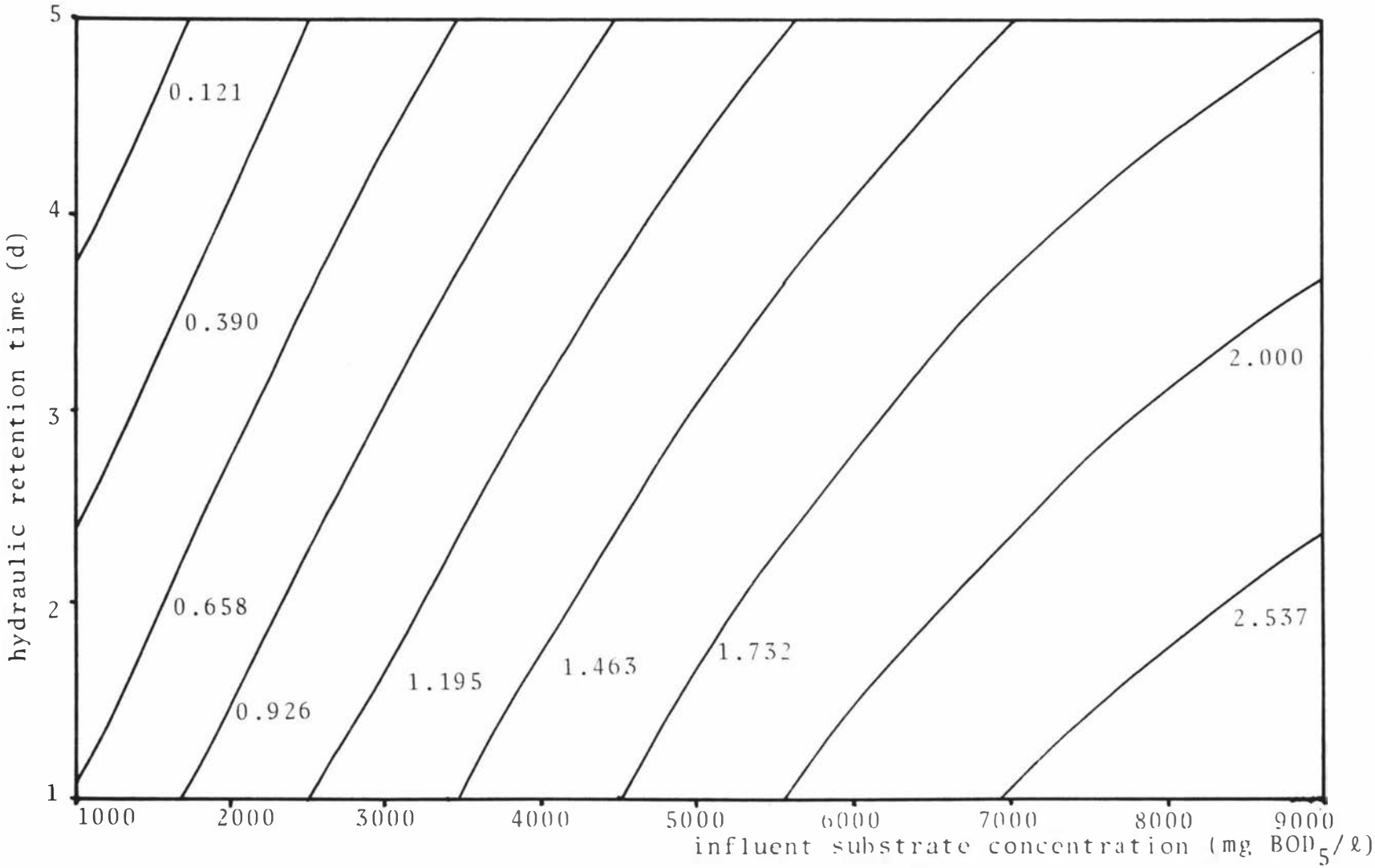


FIGURE 7.2: Contour plot of gas production model at 30°C



Max. 2.805 ℓ/ℓ
at 9000 mg BOD_5
1.0d HRT

FIGURE 7.3: Contour plot of gas production model at 35°C.

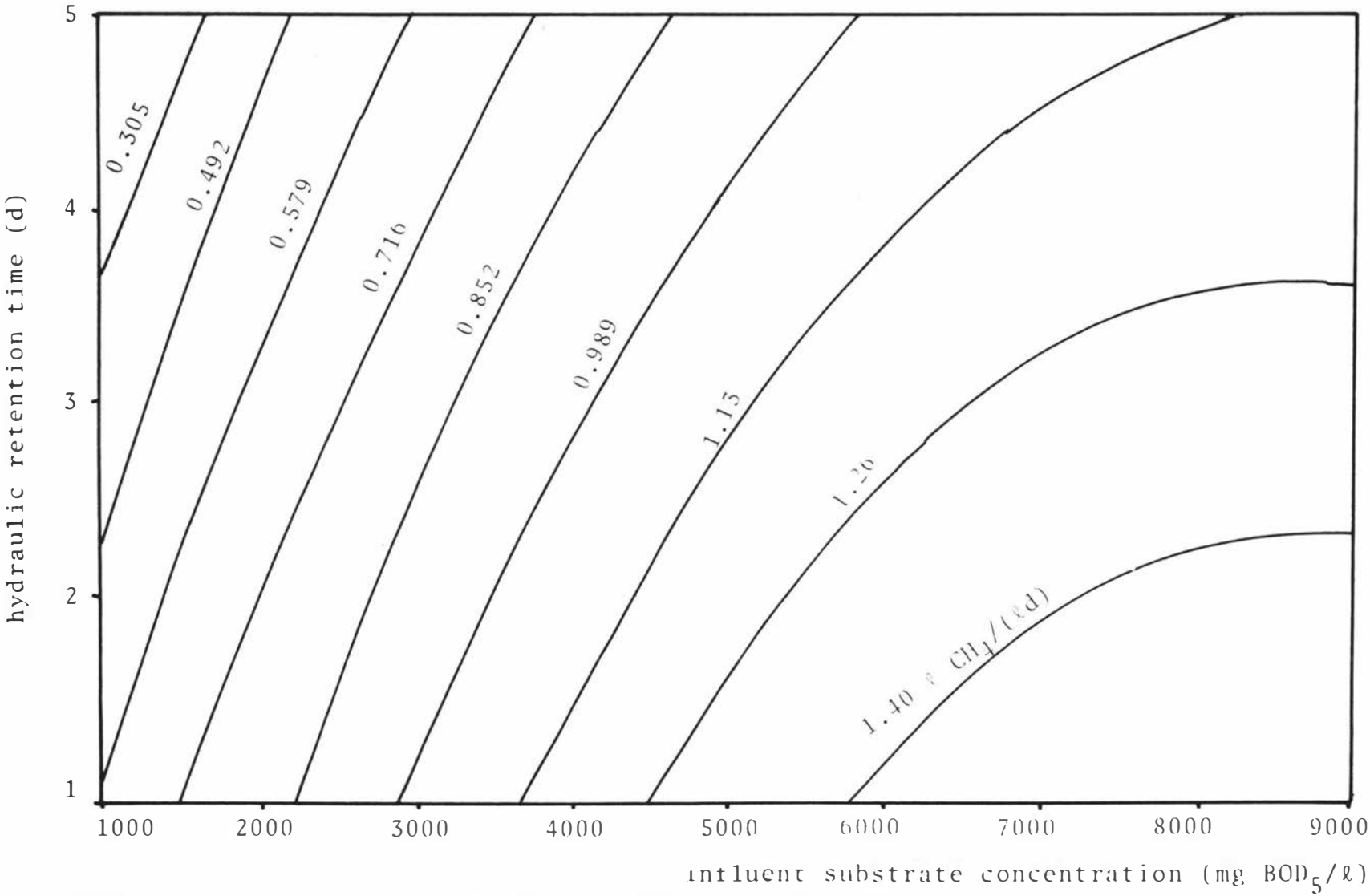


FIGURE 7.4: Contour plot of methane production model

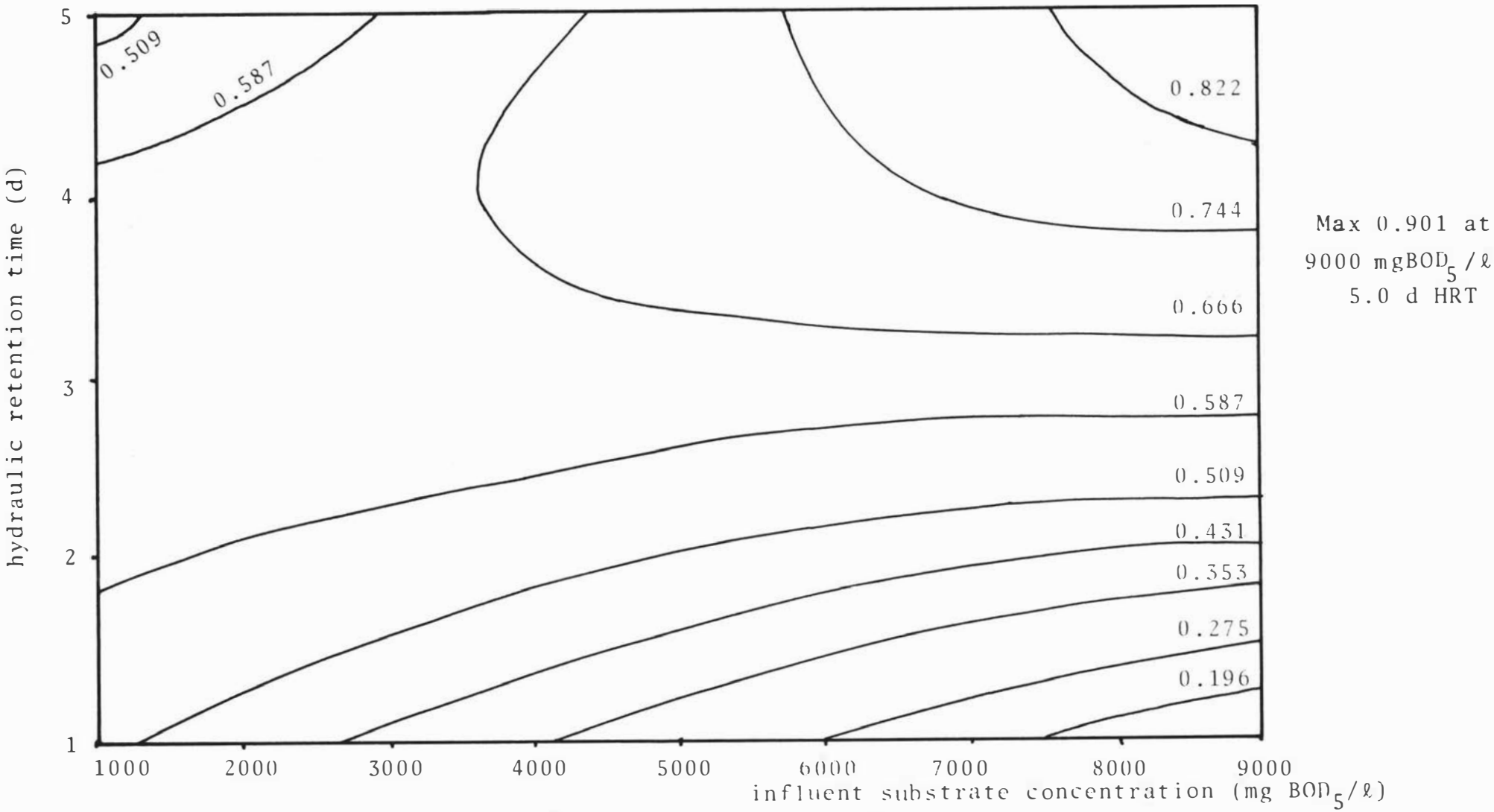


FIGURE 7.5: Contour plot of FTCM model for AF1 at 30°C

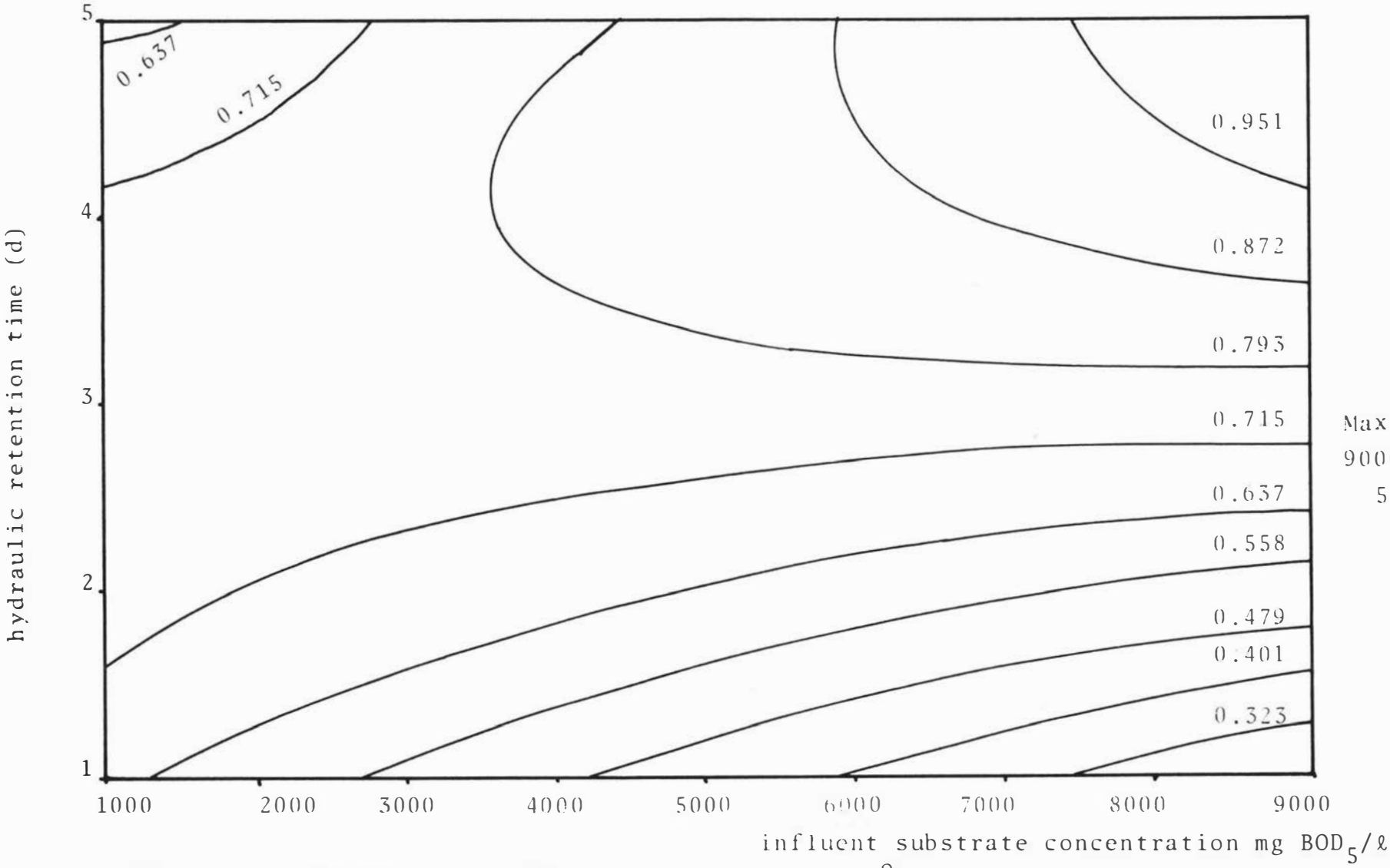


FIGURE 7.6: Contour plot of FTCM model for AF1 at 35°C

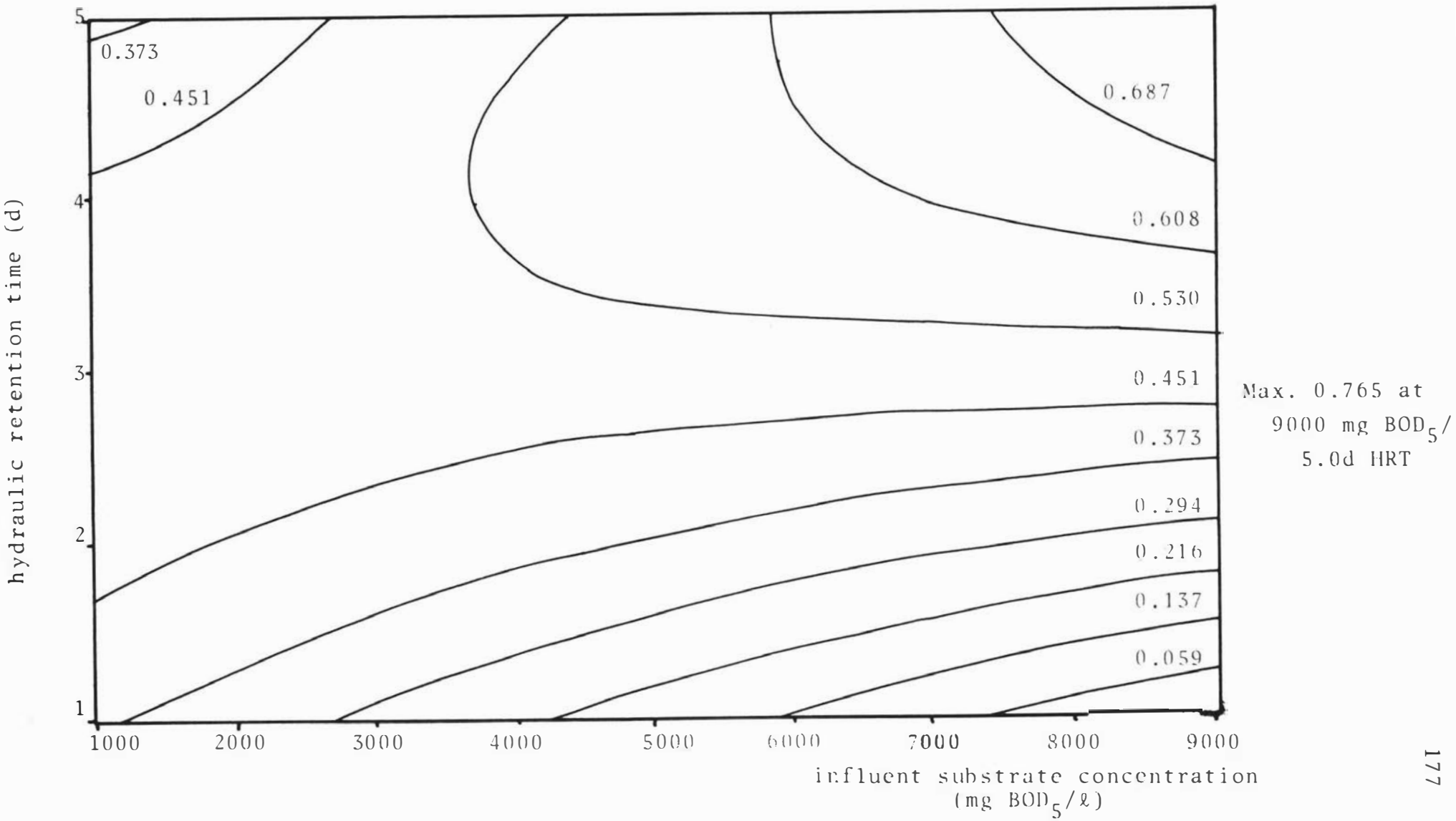
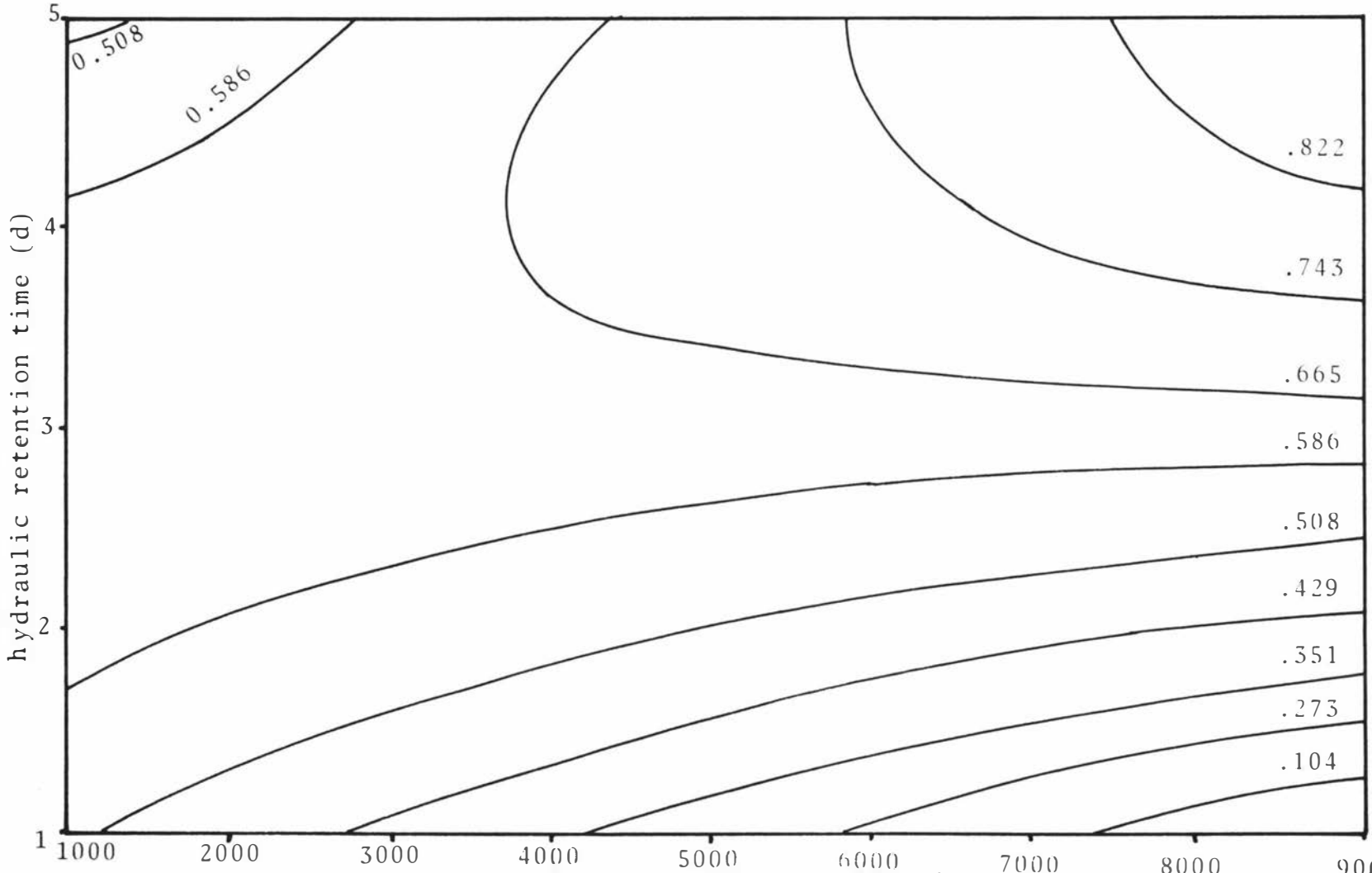


FIGURE 7.7: Contour plot of FTCM for AF2 at 30°C



Max. 0.900 at
9000 mg BOD_5/ℓ
5.0d HRT

FIGURE 7.8: Contour plot of FTCM for AF2 at $35^{o}C$

of a saddle point. The predicted maximum operating responses are 0.901, 1.03, 0.765 and 0.900 for filter units 1 and 2 at 30 and 35°C respectively.

These COD to methane conversion efficiencies have been computed using a theoretical maximum based on COD reduction when all the COD is convertable to methane. Since not all COD is carbonaceous a resistant fraction will be evident and this will vary from one waste stream to another. Chen and Hasimoto (1978) have suggested that the actual maximum possible conversion efficiency is the intercept on a plot of actual methane produced per kg of COD against reciprocal retention time. Such a plot for the data of the present study yield a maximum value of 0.274 m^3CH_4/kg COD with a correlation coefficient of r = 0.884 and one data point significantly different (95% CI) than the value predicted by the linear regression line. When that data point was omitted the maximum was 0.281 m^3CH_4/kg COD (r = 0.950) which is 80 percent of the theoretical maximum. Conversion efficiencies in this study are thus better than at first appear,as are percentage COD removals by the same factor.

7.3.1 Maximal Operating Conditions

Two additional experimental runs were carried out to test the repeatability of the previous runs. It was decided that these runs should be carried out at the predicted maximum operating points for both COD removal and gas/methane production, that is 1d HRT and 9000 mg BOD_5/ℓ. A temperature of 30°C was chosen as there appeared to be little benefit from the higher temperature used in previous design and another temperature condition could not be tested as its use could negate the repeatability aspect.

In addition to the above conditions, the effect of reducing the soluble sulphide concentration was also investigated by adding ferric chloride to the feed of the smaller filter. This aspect has been discussed in the preceding chapter. Table 7.6 summarises the results of the repeated runs as

TABLE 7.6: Observed, Predicted and Re-evaluated Values for the Regression Model Parameters

Parameter	Observed (1)	Predicted	Re-observed AF1	Re-observed AF2	Re-observed Average	Differences (%) obs.→ pred.	Differences (%) rerun → pred.
COD removal rate kg/m^3d	16.08	15.66 ± 1.98 (2)	16.373	15.88	15.979	+ 2.7	+ 2.0
Gas production ℓ/ℓd	3.110	2.793 ± 0.409	3.247	2.865	3.056	+ 4.6	+ 2.6
Methane production ℓ/ℓd	1.552	1.549 ± 0.210	1.552	1.329	1.441	+ 0.2	- 7.0
FTCM	0.246	0.252 ± 0.092	0.270	0.243	0.257	- 2.0	+ 2.0

NOTES: (1) Results from relevant experimental design run

(2) 95% confidence bounds on predicted value

they concern the predictor models

On rerun the COD removal rate was observed to be extremely close to both the original run and the predicted value, as was the conversion of COD to methane. Total gas and methane production was higher in the smaller, ferric chloride treated, filter than the larger one. For this filter the total gas production was just outside the 95 percent confidence bound of the predicted value however it was very similar to the original run. Methane production in the larger, non treated, filter was down on both the treated filter and previous run. Overall the repeatability was good to less than 5 percent of previous run and 7 percent of the predicted value.

The difference in gas and methane production between the treated and untreated filters could not be contributed to reduction of sulphide concentration since the treated filter performance showed no improvement over the previous run which had no sulphide removal treatment.

7.4 Statistical Lack of Fit of the Models

In both the COD removal rate and methane production models two of the independant variables are omitted from the response equations. By accepting the null hypothesis that the effects of these parameters, temperatures and anaerobic filter size,are not significantly different from zero the design may be viewed as a complete 3^2 factorial plus three replicate runs.

From Table 6.1 it is noted that runs 1 and 2 are replicates in respect of the two independant variables used in the model, namely influent substrate concentration and hydraulic retention time, with both variables at their low level. Similarly, runs 6 and 7 , 11 and 12,are replicates with both factors at the mid and high levels respectively.

TABLE 7.7: Replicate Data for Lack of Fit Test

COD Removal rate kg COD/m^3d	Methane Production ℓ/ℓd	Level
3.336	0.706	1
3.599	0.519	1
3.557	0.859	2
5.019	1.337	2
3.754	1.105	3
3.767	1.309	3

The data for the replicated runs has been repeated in Table 7.7 and each pair of replicates assigned a level. A oneway analysis of variance for each set of responses and the assigned levels has been performed (Table 7.8) to ascertain the amount of variance due to lack of fit using the method described by Belz (1973).

TABLE 7.8 Oneway Analysis of Variance Summary for Replicate Runs

SOURCE	d.f.	SUM OF SQUARES	MEAN SQUARE
COD removal model			
level	2	0.695	.347
error	3	1.105	.368
total	5	1.800	
Methane production model			
level	2	.4191	.2095
error	3	.1815	.0605
total	5	.6006	

Using Belz's methods, the residual variance of the regression models may be partitioned into that due to pure or experimental error and that due to lack of fit of the model, viz:

SS (lack of fit) = SS (residual) (from regression) - SS (error) (from oneway analysis of variance)

The calculations are presented in Table 7.9.

Comparison of the F values calculated in Table 7.9 with those given for the 99 and 95 percent confidence levels indicate that there is no statistically significant lack of fit in these models.

Statistical lack of fit cannot be tested for in the case of the gas production and conversion of COD to methane models as there are insufficient replicated runs to generate degrees of freedom for the significance tests.

TABLE 7.9: Lack of Fit Analysis for COD Removal and Methane Production Models

COD removal rate model

$SS_{residual}$	(table 6.2)	=	8.87	d.f. = 7
SS_{error}	(table 6.7)	=	1.105	d.f. = 3
$SS_{lack\ of\ fit}$		=		
		=	7.765	= 4

mean square lack of fit	=	7.765/4	
	=	1.941	
mean square error	=	8.87/7	
	=	1.27	
F	=	1.941/1.27	
	=	1.523	

Methane production model

$SS_{residual}$	(table 6.4)	=	.1841	d.f. = 8
SS_{error}	(table 6.7)	=	.1815	d.f. = 3
$SS_{lack\ of\ fit}$		=	.1841-.1815	d.f. = 8-5
		=	.0026	= 5

mean square lack of fit	=	.0026/5	
	=	.0005	
mean square error	=	.1841/8	
	=	.0230	
F	=	.0005/.0230	
	=	.022	

$F_{.01,\ 4,\ 7} = 5.52$; $F_{.05,\ 4,\ 7} = 4.12$

$F_{.01,\ 5,\ 8} = 6.63$; $F_{.05,\ 5,\ 8} = 3.69$

7.5 Trends in the Experimental Data

The difference between the observed and predicted values of Y in the regression equations are presented in Table 7.10. The column headed residual is the difference Y (observed) - Y (predicted). The residuals are standardised by dividing each residual by the estimated standard deviation of that residual, and these values are tabulated in column four of Table 7.10.

The standardised residuals are plotted against the independent variables, influent substrate concentration and hydraulic retention time, and also temporal run order in the subsequent Figures 7.9 to 7.18.

Inspection of these figures indicates that neither of the independent variables exerted a consistent effect on the residuals, the spread of points about the zero residual level was different in each model for both parameters. Large deviations from predicted behaviour, measured as standardised residual greater than 2.0 exist in run 10 of the COD removal rate model and run 8 of the conversion of COD to methane model. The input conditions for these runs were 9000 mg BOD_5/ℓ-3d and 5000 mg BOD_5/ℓ-5 days respectively. It was not possible to assign a cause for these large deviations as the other models did not exhibit similar deviations. In fact both these runs were carried out on the same anaerobic filter unit. However, in temporal run order they were runs nine and six respectively so a temporary filter abberation cannot be implicated either.

Significant time trends in the data would be indicated by non-random scatter or residual values about zero when plotted against run order (Figures 7.9 to 7.12)

TABLE 7.10: Observed and Predicted Values of Y in the Response Equations and the Residuals

COD removal rate model

Y. Obs.	Pred. Y Value	Residual	St. Res.
3.34	3.98	-0.65	-0.76
3.60	3.98	-0.39	-0.45
1.32	0.37	0.95	1.08
0.59	1.57	-0.98	-1.72
10.43	9.82	0.61	0.63
3.56	3.78	-0.22	-0.22
5.02	3.78	1.24	1.28
3.05	2.55	0.51	0.53
16.09	15.66	0.43	0.75
5.20	7.18	-1.98	-2.24
3.75	3.52	0.23	0.27
3.77	3.52	0.25	0.29

Total gas model

Y. Obs.	Pred. Y Value	Residual	St. Res.
0.845	0.696	0.149	0.71
0.599	0.696	-0.097	-0.46
0.409	0.520	-0.111	-0.50
0.173	0.114	0.059	0.32
1.770	2.127	-0.357	-1.79
1.296	1.492	-0.196	-0.88
2.010	1.721	0.289	1.30
1.350	1.086	0.264	1.33
3.110	2.793	0.317	1.72
2.153	2.159	-0.006	-0.03
1.640	1.753	-0.113	-0.54
1.784	1.982	-0.198	-0.95

TABLE 7.10 Con'd

Methane production model

Y. obs.	Pred. Y Value	Residual	St. Res.
0.706	0.577	0.129	1.03
0.519	0.577	-0.057	-0.46
0.335	0.374	-0.039	-0.30
0.138	0.171	-0.033	-0.29
1.343	1.317	0.027	0.22
0.859	1.114	-0.255	-1.94
1.337	1.114	0.223	1.69
0.917	0.911	0.005	0.05
1.552	1.549	0.003	0.03
1.214	1.346	-0.132	-1.01
1.105	1.143	-0.038	-0.30
1.309	1.143	0.166	1.33

FTCM Model

Y.obs.	Pred. Y Value	Residual	St. Res.
0.541	0.520	0.021	0.95
0.369	0.391	-0.022	-0.74
0.651	0.640	0.011	0.32
0.600	0.634	-0.034	-1.13
0.331	0.324	0.007	0.20
0.618	0.634	-0.016	-0.47
0.682	0.640	0.042	1.22
0.772	0.700	0.072	2.13
0.246	0.252	-0.006	-0.19
0.597	0.634	-0.037	-1.07
0.756	0.773	-0.017	-0.58
0.887	0.908	-0.021	-0.95

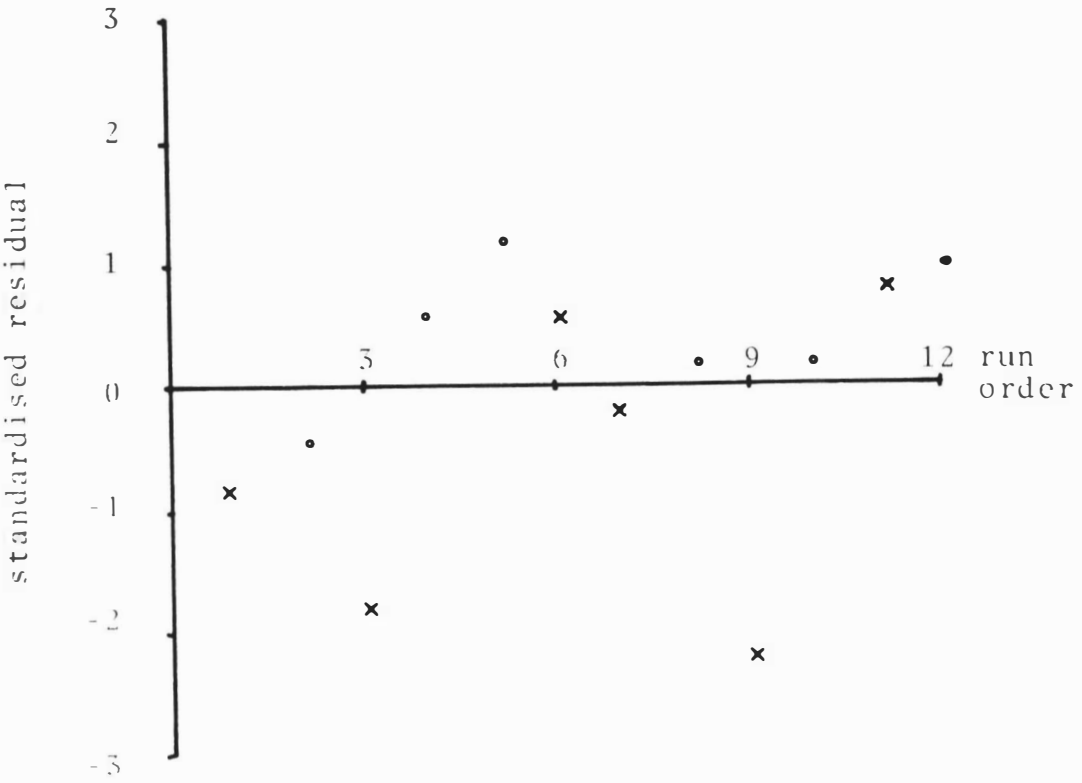


FIGURE 7.9: Plot of standardised residuals from COD removal rate model against temporal run order

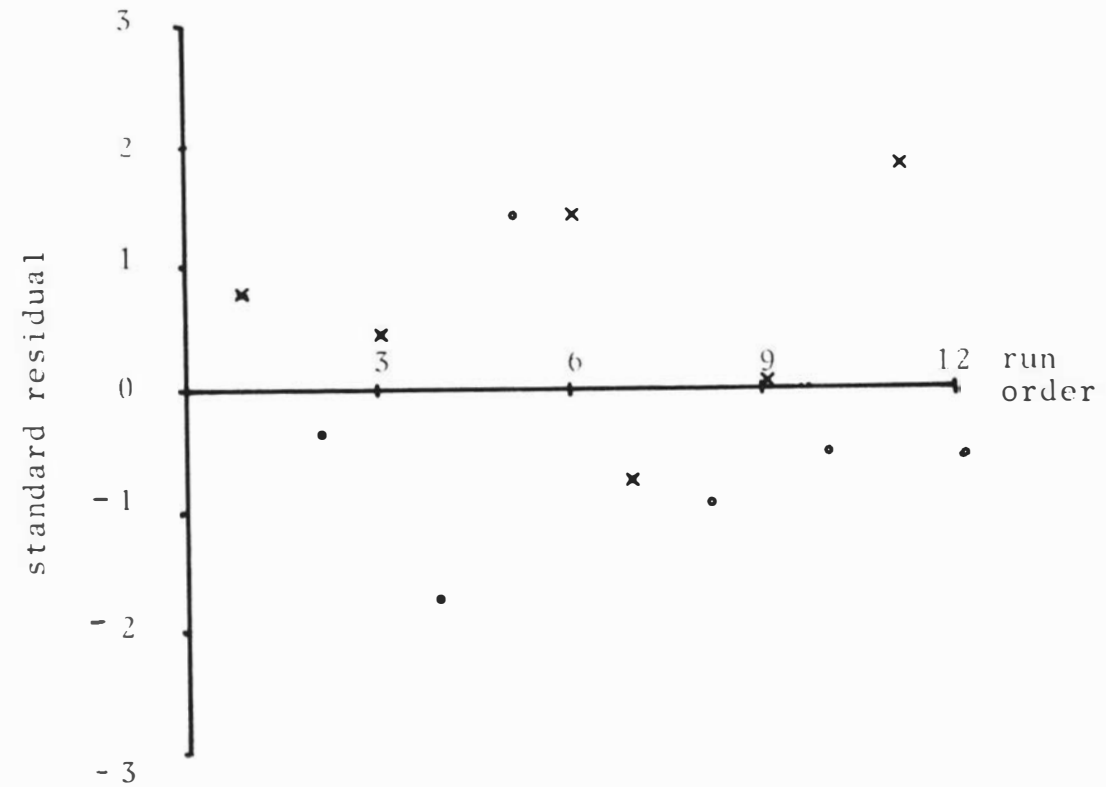


FIGURE 7.10: Plot of standardised residuals for total gas production model against temporal run order

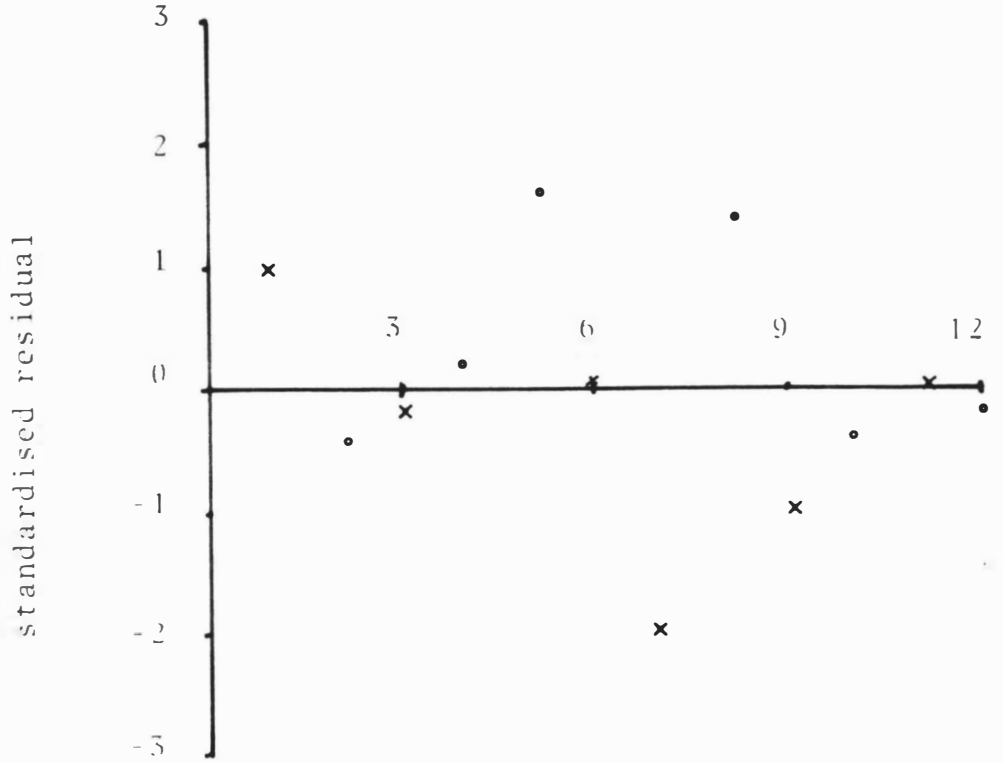


FIGURE 7.11: Plot of standardised residuals for methane production model against temporal run order

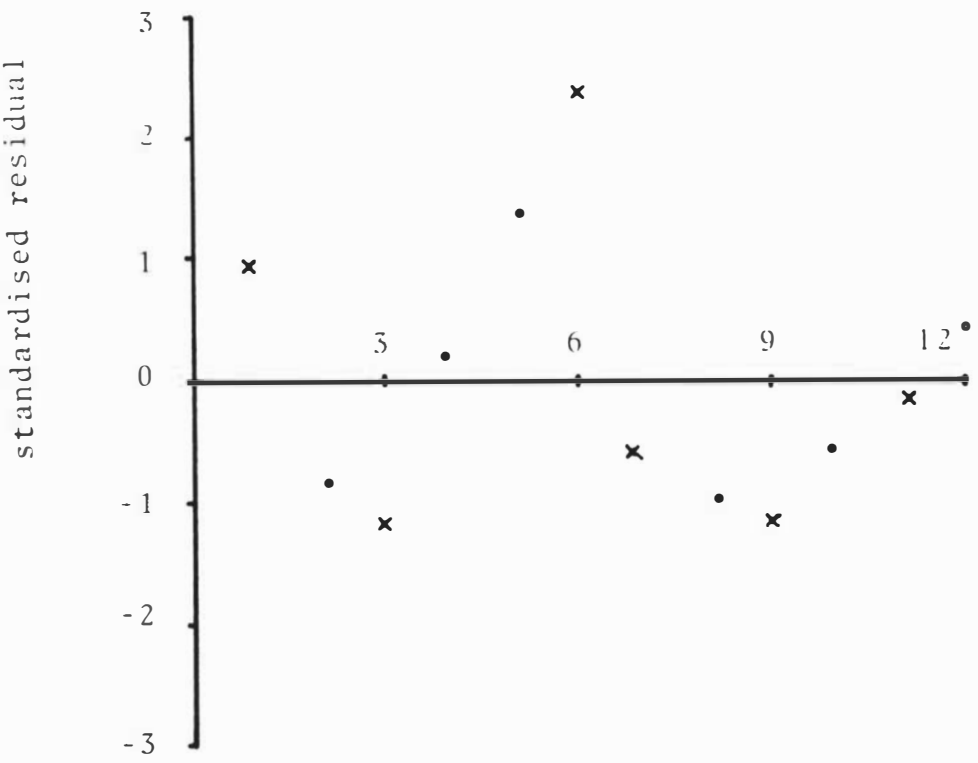


FIGURE 7.12: Plot of standardised residuals for FTCM model versus temporal run order

Examination of the residuals is important as large deviations from predicted behaviour can be picked up and often shows where mistakes have been made, for instance in measurements of experimental parameters, or assumptions made in the model are not valid or the wrong model has been chosen. The residuals plots are presented in Appendix 2 for the main input parameters.

Of particular importance in this study was the possible effects of a significant time trend exerting an influence on the results. Because the experimental design was carried out over an extended period of time due to the nature of the system required long periods of build up to new operating conditions. The spread of residuals above and below zero in the following four plots indicate that there was no immediately obvious trend in the residuals over the experimental time period. Likely patterns are fanning out (or in) of residuals indicating the model was worse (or better) at predicting earlier runs than later. It is possible that for the conversion of COD to methane model residuals (Figure 7.11) the larger filter followed a cyclic pattern in runs order 1-3 and 4-6 and that in each cycle, conversion efficiency improved over the previous runs. This is a possibility as the biomass could have become, say, better acclimated as time went by but one would expect this to occur over the entire run number and not to cycle.

One may then conclude that the experimental design chosen was not compromised in its assumption of no unaccounted for or system bias over the experimental period.

7.6 The Use of Waste Treatment Models

When the basic principles of a process are simple and well established a theoretically based model can be formulated. This is often the case in chemical processes but not usually

applicable to the highly variable conditions found in waste treatment processes. The alternative is to set up an empirical model in the form of a mathematical or graphical presentation which is not necessarily related to the theory of operation but gives a good description of the observed behaviour (Tebbutt, 1978). The mathematical model most often used to describe anaerobic digestion studies is the simplified mass balance model proposed by Eckenfelder & O'Connor (1961) and also Earle (1962). For a completely mixed system at steady state, the effluent concentration is related to hydraulic retention time in equation 7.11.

$$\frac{S_e}{S_o} = \frac{1}{1 + Kt} \qquad (7.11)$$

where S_e = effluent substrate concentration
S_o = influent substrate concentration
K = substrate removal coefficient
t = retention time

This and other kinetic models, such as the Monod growth model, require that substrate removal coefficients be defined in terms of the rate limiting substrate for digestion to proceed. Many anaerobic digestion studies are carried out using synthetic, chemically defined substrates in which the rate limiting components are readily identified. In the study of real industrial waste water streams of complex and often variable nature gross subtrate concentration measurement by COD or volatile solids is essential (Chen & Hashimoto, 1978). The fact that not all COD is biologically degradable and contains a resistant fraction has been accounted for in the mass balance model by altering equation 7.11 to equation 7.12 (Eckenfelder & Ford, 1971),

$$\frac{S_o - S_e}{S_e - S_r} = Kt \qquad (7.12)$$

where S_r = resistant COD concentration

The application of the mass balance model of equation 7.11 to the data of the present study is presented in figure 7.13 which is a plot of $(S_o/S_e)-1$ against time for each of the three levels of influent substrate concentration.

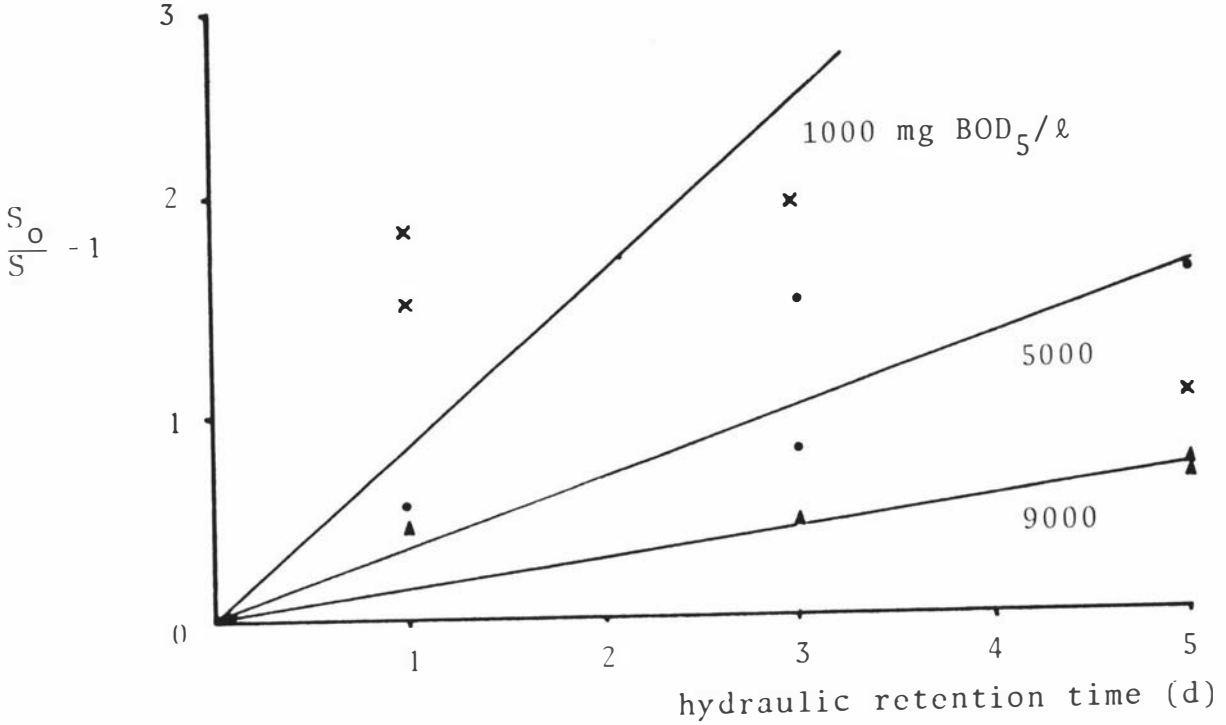


FIGURE 7.13: Mass balance model applied to data of the present study

There appears to be reasonable fit at the higher loading rates, however at the lowest concentration of 1000 mg BOD_5/ℓ much scatter is evident and the point at 5d HRT is greatly out of line. This latter point has been ommitted in calculation of the K values which were at 1000 mg/ℓ 0.87 d^{-1}, 5000 mg/ℓ 0.36 d^{-1} and 9000 mg/ℓ 0.15 d^{-1}. Statistics for these regression lines were r^2 = 0.82, 0.94 and 0.90 respectively. It is not felt that this model adequately fits the data in that from Figure 7.13 it is likely that there is rapid stabilisation of readily degradable portion of the waste at short retention times of less than about 1d and that K then decreases as the more resistant material is degraded. To take into account the effect of a resistant fraction of COD, equation 7.12 is used for the data of the present study and illustrated in Figure 7.14. Rearrangement of equation 7.12 into the form of equation 7.13 was used.

$$S_e = \frac{S_o - S_e}{Kt} + S_r \qquad (7.13)$$

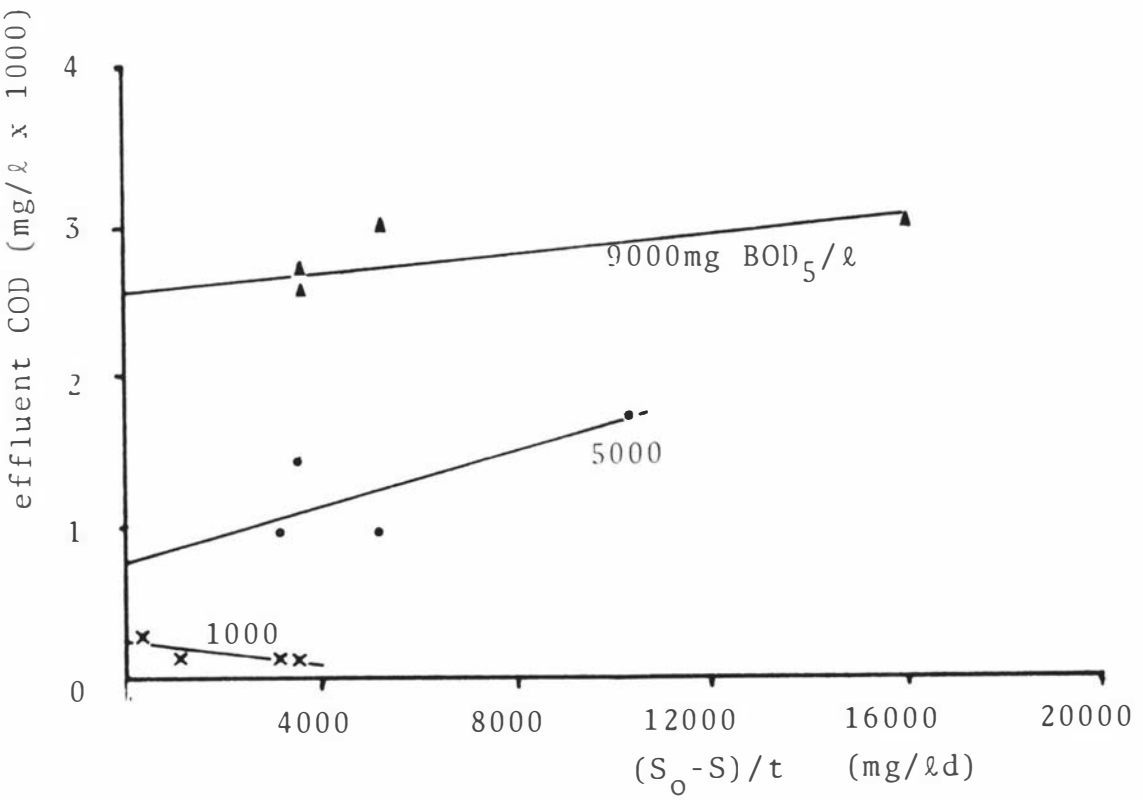


FIGURE 7.14: Mass balance model incorporating resistant COD fraction applied to data of the present study

Linear regression lines were calculated for each data group figure 7.14 and are presented in table 7.11.

TABLE 7.11: Regression Analysis of Plots in Figure 7.14

S_o mg BOD_5/ℓ	mean influent COD mg/ℓ	K d^{-1}	S_r mg COD/ℓ	r^2
1000	5660	*	2620	0.14
5000	25320	1.1	7370	0.49
9000	45980	3.4	26660	0.15

* negative slope of least squares line

From the r^2 values in Table 7.11 it is evident that the applied analysis does not fit the data well. The resistant fraction also varied between 0.46, 0.58 and 0.29 at the low, high and mid range influent substrate concentrations respectively. All the feed material was diluted from stock material of average BOD_5 14250 mg/ℓ and the apparent variations in resistant fraction resulting from inadequate preparation is highly unlikely since the runs were carried out on a randomised basis over the experimental period to eliminate such bias. Although the use of the mass balance equations is widespread in the literature, their use is probably limited to narrow concentration ranges and best applicable to synthetic substrates in which the rates limiting concentration can be readily identified. Switzenbaum (1978) found equation 7.11 adequately described performance over the range of 300 to 600 mg COD/ℓ using a synthetic waste comprising glucose plus yeast extract and salts and detention times of 0.33 to 6 hours. Little, if any of the COD of this waste would have resisted biodegradation. On the other hand, in trials assessing the amenability of domestic refuse to anaerobic digestion K was found to vary between zero and 0.769 d^{-1} in a non-uniform manner over a range of HRT of 1.5 to 14.6 d (Pffefer, 1978). This was concluded to be due to the complex nature of real waste streams and further it was doubted whether any kinetic model could be adequately fitted to the data for the same reason.

In the realm of anaerobic filtration studies, several models have been proposed to predict filter performance. These range in complexity between complex considerations based on the theories of biofilm kinetics (Jennings *et al*, 1976) to Monod based kinetic appraisals (Young & McCarty, 1967) and first order kinetics (Mueller and Mancini, 1975). For use in a design situation a performance model need to be as simple as possible, and also to reflect the variable nature of the system. The real situations encountered in the treatment of complex wastes by mixed populations and the limitations of applying pure culture kinetics those have already

been noted (Downing, 1966). The possibility that the rate limiting substrate-organism combination varies under different loading conditions has been propounded (Pffefer, 1978). This can also be adduced from the variation in estimates of kinetic parameters of greater than an order of magnitude in reviews of literature for models such as Monod based kinetic analyses of anaerobic digestion (Zehnder *et al*, 1981).

Perhaps a unifying concept for anaerobic digestion and indeed any biological process will only be achieved when the processes are reduced to a molecular level and a, necessarily, vast model requiring complete characterisation of both substrates and organisms (including their ability to mutate) is produced. Until then it is not unlikely that simplistic empirical models will have to suffice.

The use of statistically based regression models is therefore seen as a viable alternative to chemical and pure-culture-biological kinetics for the modelling of anaerobic filtration. There does exist a class of problems in which regressions do exist, but there is no functional relationship between variables, especially in biological systems (Davies, 1954). This could well be the case when using gross substrate measurements, such as COD, in biological waste treatment studies as they are of practical necessity.

When only a limited number of experimental trials are able to be carried out the statistically based experimental design generates much more data than the classical one variable at a time approach. In addition while many models may not be linear in the process variables, regression analysis allows fitting of transformed, coded variables to produce models which can easily be analysed as the equations become linear in their parameters and interactions between variables can be quantified. While the present study has only isolated the parameters having the greatest effect on anaerobic filter performance, from the treatment of a high strength industrial

waste, those parameters have been used, to quantify that performance in equations which may be used to predict the performance of a real waste treatment system over a greater range of process variable levels than previously tested The technological significance of the equations will be demonstrated in the following chapter on the basic design of an on site effluent pre-treatment process.

CHAPTER EIGHT

ON-SITE PRETREATMENT OF YEAST PLANT EFFLUENT: AN ANALYSIS OF PLANT SIZE OPTIONS

CHAPTER EIGHT

ON-SITE PRETREATMENT OF YEAST PLANT EFFLUENT: AN ANALYSIS OF PLANT SIZE OPTIONS

A factory is prodicing up to five batches of bakers' yeast per week. The amount of yeast propagation broth and yeast cream wash water produced is presented in Table 8.1. For the purposes of the analysis the items listed in Table 8.1 are considered to contribute 100 percent of the waste water oxygen demand and 40 percent of the total volume. It is also assumed that the sludge produced by the molasses clarification will be disposed of separately in a solid form and not flushed to waste. The remaining waste water volume will therefore consist primarily of fermenter cooling water and washings from the plant and will not contribute significantly to the oxygen demand.

TABLE 8.1: Factory Discharges of Spent Media and Yeast Cream Wash Waters.

Item	Volume	BOD_5
	m^3/week	mg/
Spent media	165	14250
1st wash water	90	5000
2nd wash water	125	1250
3rd wash water	110	500*
TOTALS	490	6160+

* estimated
\+ weighted for volume

The total BOD_5 discharged from the plant is therefore 3020 kg/week. The waste streams itemised in Table 8.1 are readily isolated, from the other liquid wastes from the

factory and can be chanelled to a treatment facility located on-site.

The treatment plant would consist of an equalising tank, a recirculated anaerobic filter and a sedimentation vessel. Alternatively, provision could be made for sedimentation within the filter body. The treated effluent would be mixed with the remaining plant waste water prior to discharge to the municipal sewer.

The charge levied for the acceptance of the waste water, by the local authority, is calculated from equation 8.1.

$$\$/\text{year} = \frac{V}{65}\left(1.2+\frac{0.84S}{350}+\frac{0.44B}{400}\right)-zR(2.67) \qquad (8.1)$$

where: V=average daily discharge (litres)
S=suspended solids content (mg/ℓ)
$B=BOD_5$ content (mg/ℓ)
z and R = rating coefficients

The total waste water, untreated, will attract a charge of $10 840 per year. This is a considerable sum and will only increase in the future, especially with respect to the coefficient relating to BOD_5, as the local authority is obliged to install more sophisticated BOD_5 treatment in their own plant. Several options are perceived for the amount of waste water to be treated on the factory site. These are itemised in Table 8.2. The BOD_5 content of the first two options are considered to be too high for treatment without some dilution, but the next three fall into the range known to be amenable to treatment by the anaerobic filter. In the following analysis these three are labelled A,B and C respectively.

TABLE 8.2: On-site Treatment Options

Stream to be treated	Volume m^3/week	BOD_5 mg/ℓ	
Spent media	165	14250	
Spent media + wash 1	255	11000	
Spent media + washes 1&2	380	7800	A
Spent media + all washes	490	6160	B
Total waste water	1225	2460	C

The expected operating conditions and some economic estimates for options A,B and C are presented in Table 8.3 and the notes relating to it. For convenience, the hydraulic retention times used in the previous study have been used here as well and the subscripts refer to 1,3 and 5 d HRT respectively for the options in row 1.

The bottom line in Table 8.3 indicates that options A1 and B1 give similar cost savings when compared on an equal plant investment cost basis. A choice between them would probably depend largely on the land area available for the treatment plant, since the filter required for option B1 is about twice the size of that needed for A1.

No dollar value has been placed on the cost of filter construction but it is anticipated that long pay-back periods would be manifest due to the magnitude of the savings involved. However, cost savings are not all that is to be considered in the treatment plant. The effluent is of a lower pH than that range specified by the local authority namely 6 to 9, and the anaerobic filtration process with its attendant pH adjustment will both tend to equalise the final discharge with respect to pH and BOD_5 content; thus protecting the downstream biological treatment facilities.

TABLE 8.3: Comparison of Several Treatment Plant Options

	notes	OPTION NUMBER A1	A2	A3	B1	B2	B3	C1	C2	C3
Feed concentration										
BOD_5 mg/ℓ		7800	7800	7800	6160	6160	6160	2460	2460	2460
COD mg/ℓ		39000	39000	39000	30800	30800	30800	12300	12300	12300
Volume m^3/d		54.3	54.3	54.3	70	70	70	175	175	175
HRT d		1	3	5	1	3	5	1	3	5
Effluent COD mg/ℓ	1	26535	24245	21600	19045	17630	15075	5995	4940	4010
COD removal %		32.0	37.8	44.6	38.2	42.8	51.1	51.3	59.8	67.4
BOD_5 removal %	2	40.0	47.3	55.8	47.7	53.4	63.8	64.1	74.8	84.2
Effluent BOD_5 mg/ℓ	1	4680	4110	3448	3222	2871	2230	883	620	389
Final BOD_5 mg/ℓ	3	1450	1275	1070	1295	1150	895	883	620	389
Methane m^3/d	4	83.1	72.1	61.1	150.7	129.4	108.1	158.5	123.0	87.5
Net filter volume m^3		54.3	162.9	271.5	105.0	315.0	525.0	175.0	525.0	875.0
Gross energy in methane GJ/d	5	3.12	2.71	2.29	5.65	4.85	4.06	5.94	4.61	3.28
Net energy GJ/d	6	0.92	0.64	0.34	1.93	1.37	0.81	(3.66)	(4.99)	(6.32)
Savings due to:										
BOD_5 removal		$3315	3832	4440	3773	4202	4958	4993	5772	6456
Gas credit	7	$1310	910	485	2750	1950	1155	(5210)	(7105)	(8997)
Total		$4625	4742	4925	6125	6152	6113	(217)	(1333)	(2541)
Relative cost	8	1.00	1.93	2.63	1.49	2.87	3.90	2.02	3.90	5.30
Relative cost benefit	9	4625	2457	1872	4110	2143	1567	-	-	-

Notes relating to Table 8.3

1 Filter effluent COD or BOD_5

2 Based on the assumption that BOD_5 removal will be equal to the removal of degradable COD.

3 BOD_5 content of the waste water discharged to the municipal sewer.

4 Calculated from the methane production model of this study.

5 Based on a heating value of 37.5 MJ/m^3 for methane.

6 Net energy=70% of the energy remaining after taking into account the energy required to heat the filter feed to 30^{o} C. The estimated feed temperatures of the feed material are: 22,20 and 17^{o} C for options A,B and C respectively.

7 December 1983 prices assuming the methane will replace natural gas used by the plant.

8 Using option A1 as the base plant size then:

$$\text{relative plant cost} = \text{base cost} \left(\frac{\text{volume of plant}}{\text{volume of base}}\right)^{0.6}$$

9 Total savings/relative plant cost

CONCLUSIONS

The anaerobic filter has been shown to be a suitable process for the pretreatment of a high strength, acidic waste water srising from the production of bakers' yeast. Although the rates of removal of COD were not as high as can be achieved by some anaerobic processes, such as the upflow anaerobic sludge blanket and expanded fluidised bed, the level of sophistication of the anaerobic filter is lower. This results in reduced costs for initial construction and operation. Further, although the present application has been restricted to the treatment of bakers' yeast, there is no reason to believe the filter is not applicable to other high strength industrial wastes.

The ranges of influent substrate concentration and hydraulic retention time used were: 5500 to 47200 mg COD/ℓ (1000 to 9000 mg BOD_5/ℓ) and 1 to 5 days respectively. The consequent organic loading rates were 1.15 to 47.2 kg COD/m^3d (0.2 to 9.0 kg BOD_5/m^3d). At the lower loading rates COD removals were of the order of 65 percent and these decreased steadily,as the loading rate was increased, to 34 percent at the highest loading rate tested. These values are in terms of total COD of which 80 percent was found to be degradable. The mass removal rates of COD per unit volume of the digester were found to increase with increased loading rate, with the rate of increase falling at the highest loading rates. There was also a large reduction in methane at the high loading rates, especially at shorter hydraulic retention times.

Because of a lack of fit found with the mechanistic models used to describe the anaerobic filter, an experimental approach based on a statistical design was used which lent itself to the generation of regression models. Two process responses, namely COD removal rate and methane production were found to be able to be described in terms of the influent substrate concentration and hydraulic retention time in linear or quadratic forms. Total gas production required the addition of a temperature term and the conversion of COD to

methane also required a term accounting for the anaerobic filter used. The equations described between 92 and 97 percent of the observed variance in the experimental trials over the range of conditions studied.

The anaerobic filter system was found to be experimentally stable and the results statistically consistent over the applied changes in the operating conditions, for a period of almost three years. This reliability gives a confidence in the use of the regression models to predict the operating performance in the real plant situation.

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APPENDIX 1: Regression Data for Effluent COD and Percent COD Removal

TABLE A1

Least squares analysis of effluent COD data.

Predictor	t ratio
Constant	24.20
ISC	18.43
HRT	-2.52
$(ISC)^2$	2.65
(ISC) (HRT)	1.60

Coefficient of determination $r^2 = .981$

run	std residual
5	1.88

TABLE A2

Least squares analysis of % COD removal data

Predictor	t ratio
Constant	19.38
ISC	-4.07
HRT	0.98
AF	0.40
(ISC) (HRT)	0.86

Coefficient of determination = $r^2 = .716$

run	std residual
4	-1.86

APPENDIX 2: Residuals plots

A. COD removed model

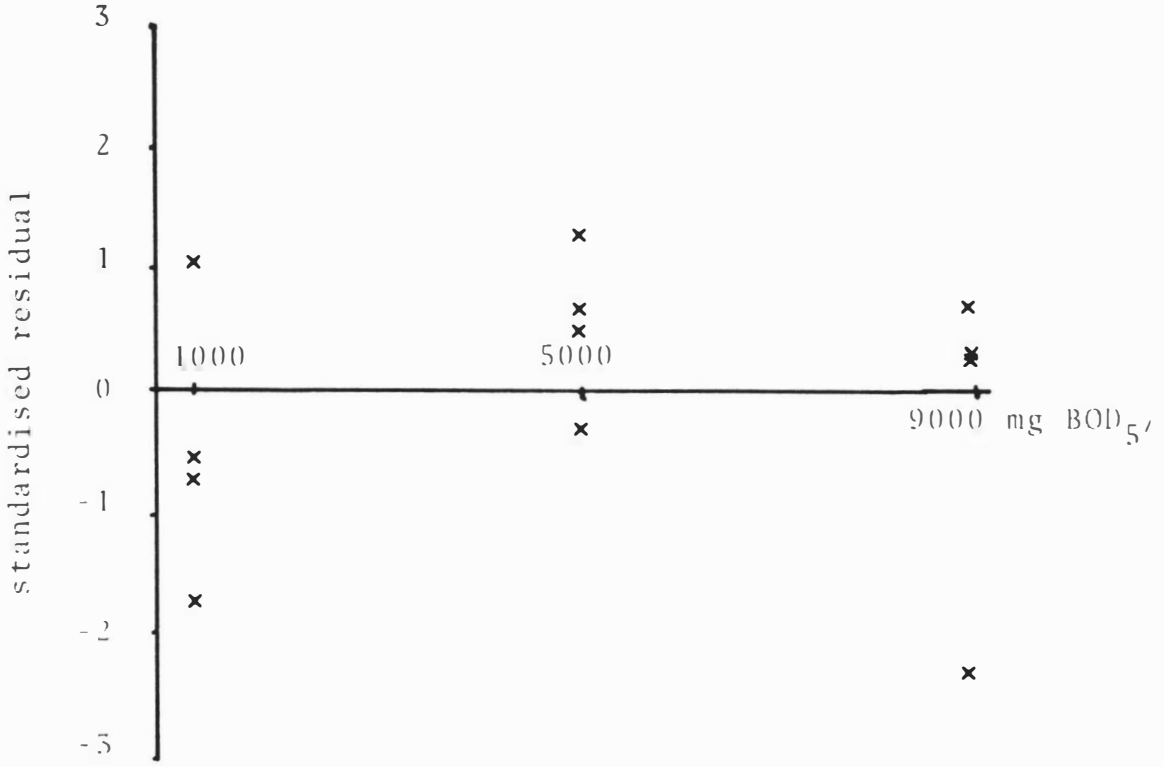


FIGURE A1: Standardised residual and influent substrate concentration

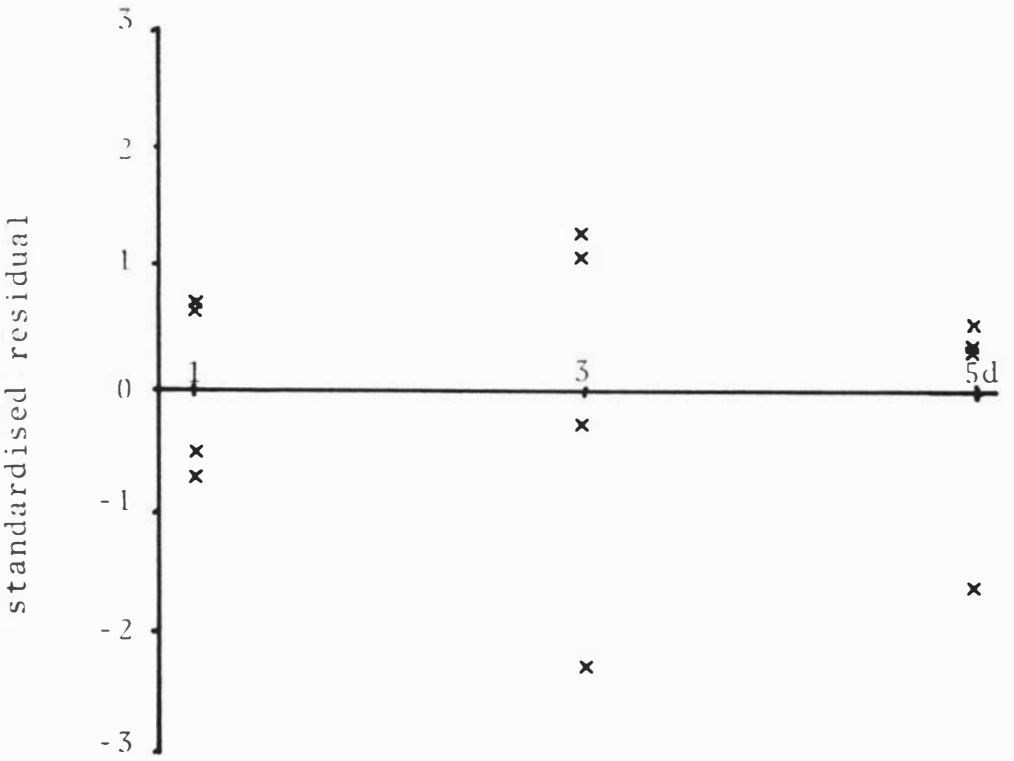
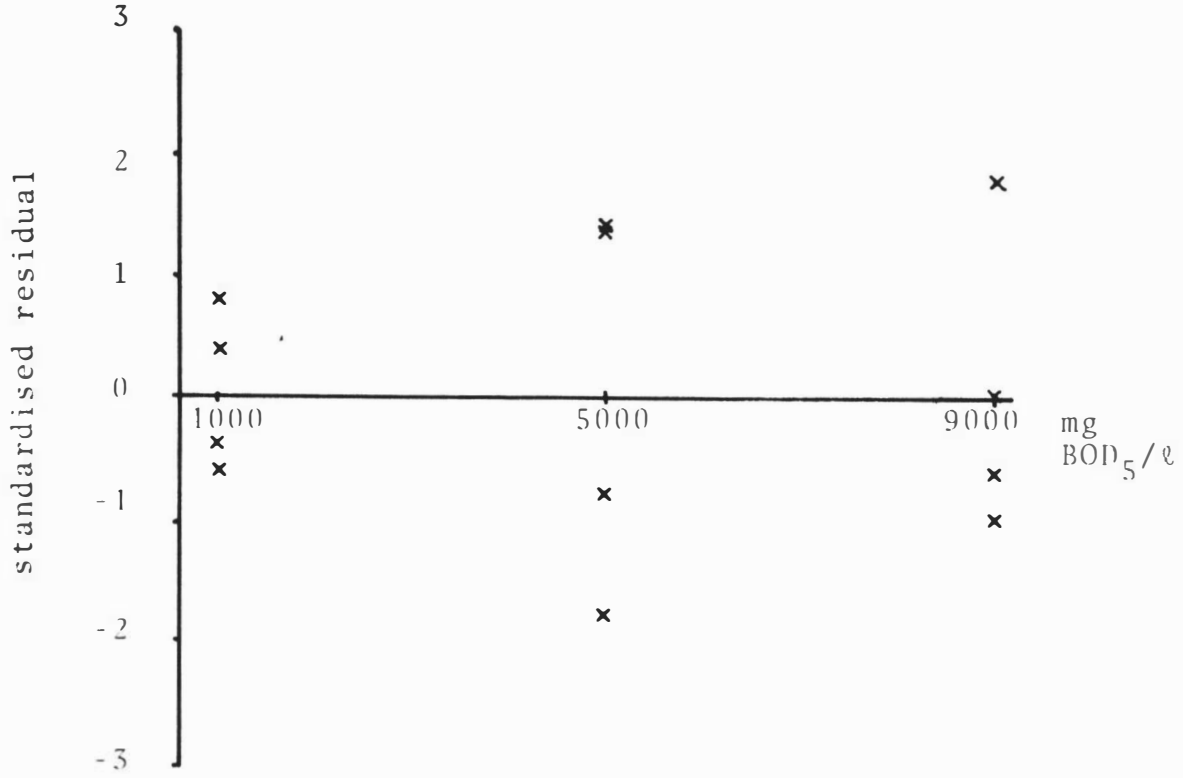
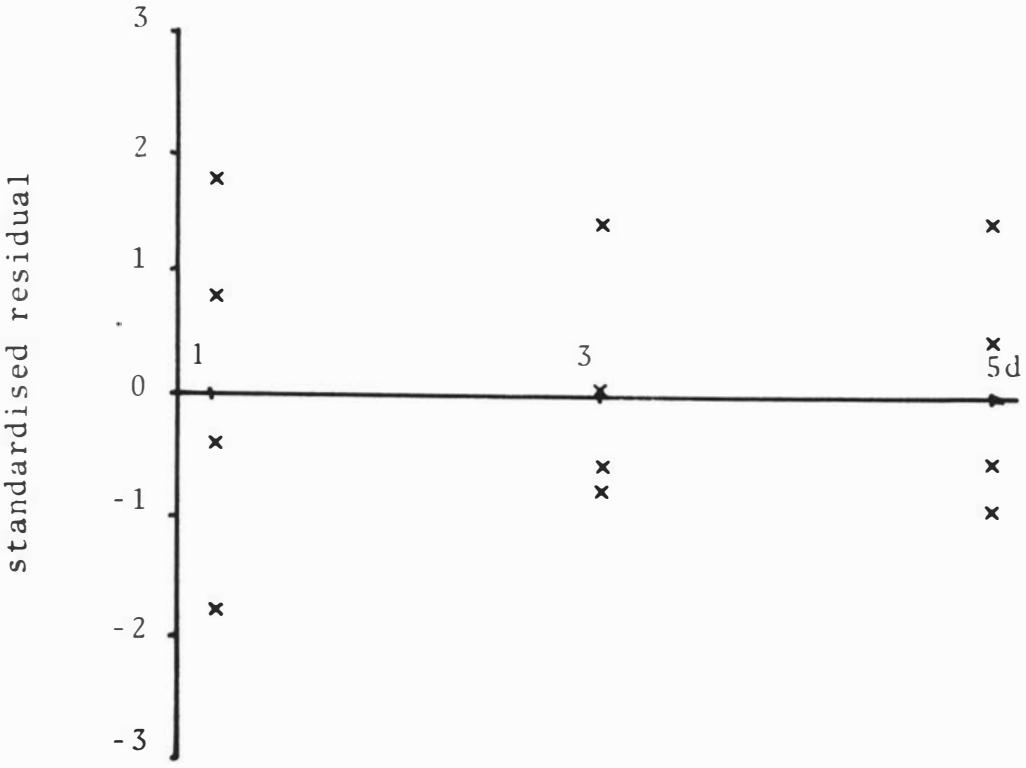


FIGURE A2: Standardised residual and hydraulic retention time



GURE B1: Standardized residual and influent substrate concentration



GURE B2: Standardized residual and hydraulic retention time

C. Methane production model

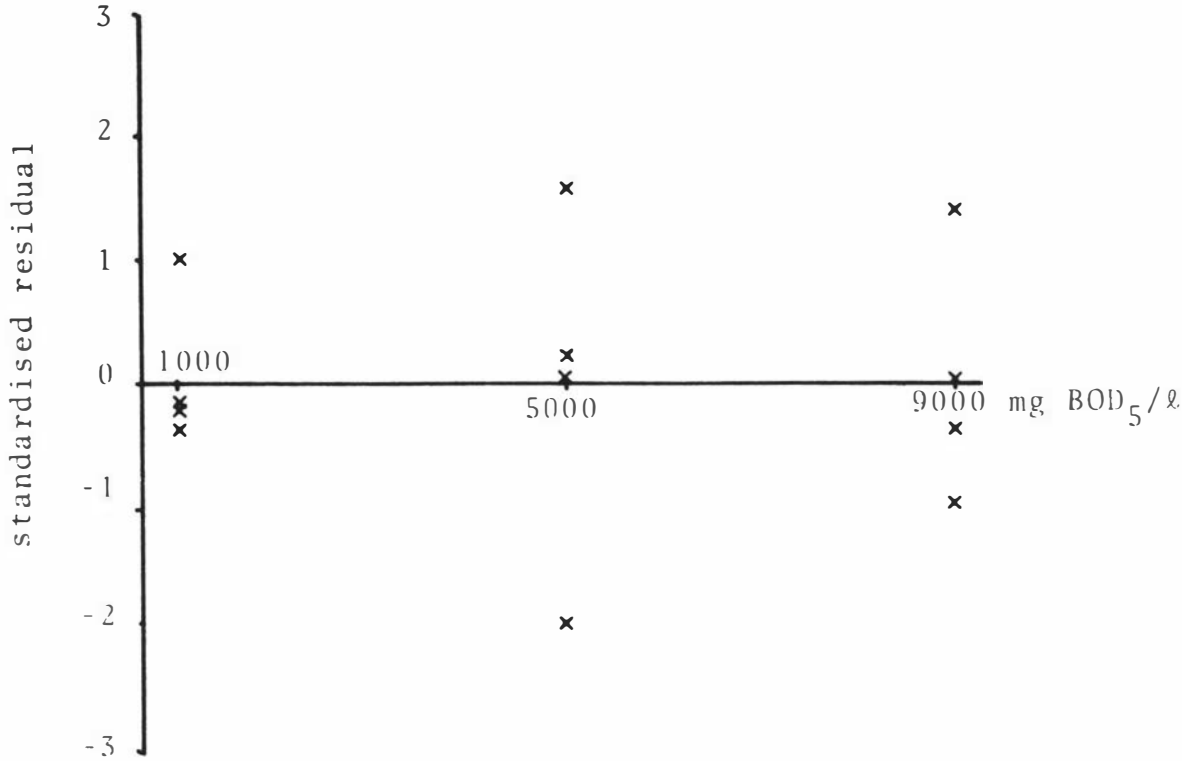


FIGURE C1: Standardised residual and influent substrate concentration

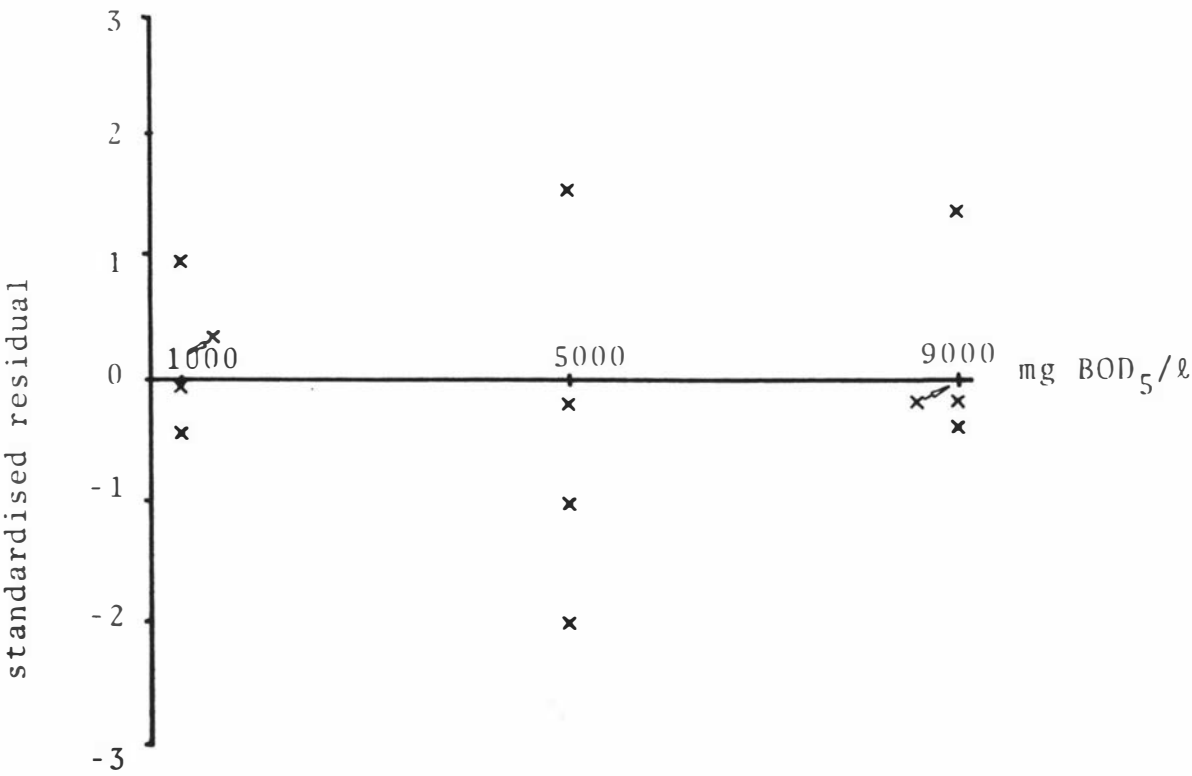


FIGURE C2: Standardised residual and hydraulic retention time

D. FTCM Model

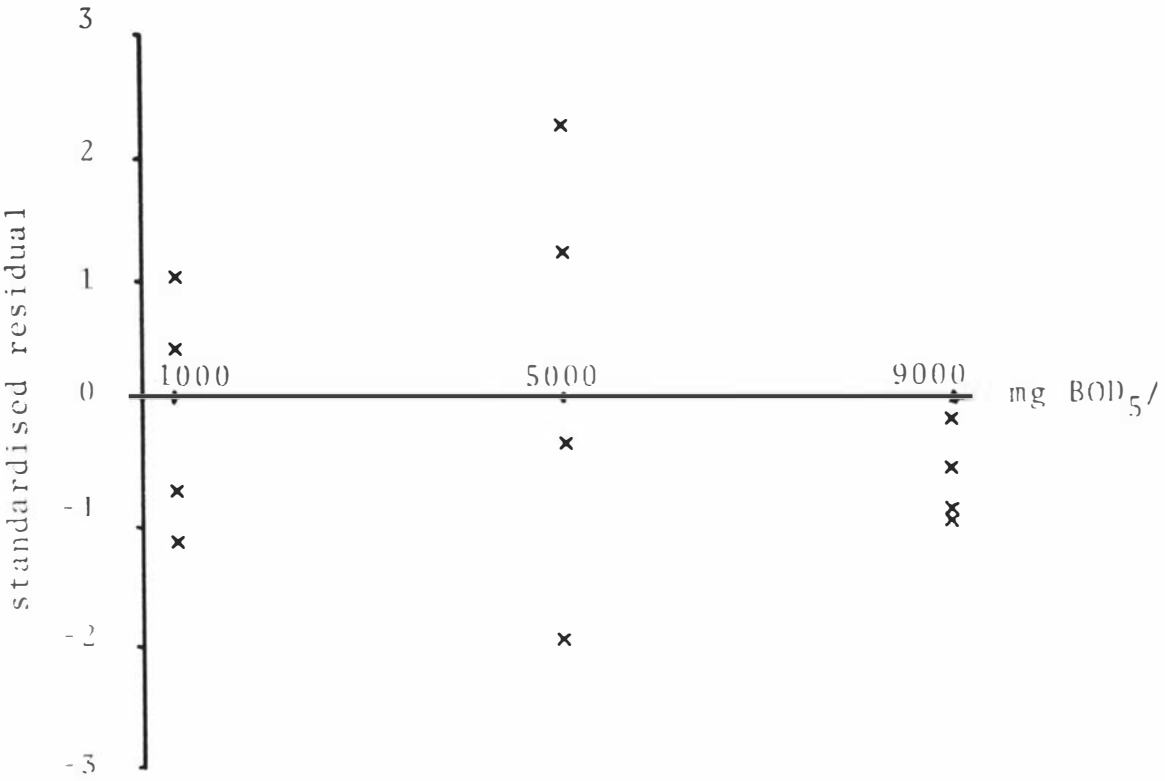


FIGURE D1: Standardised residual and influent substrate concentration

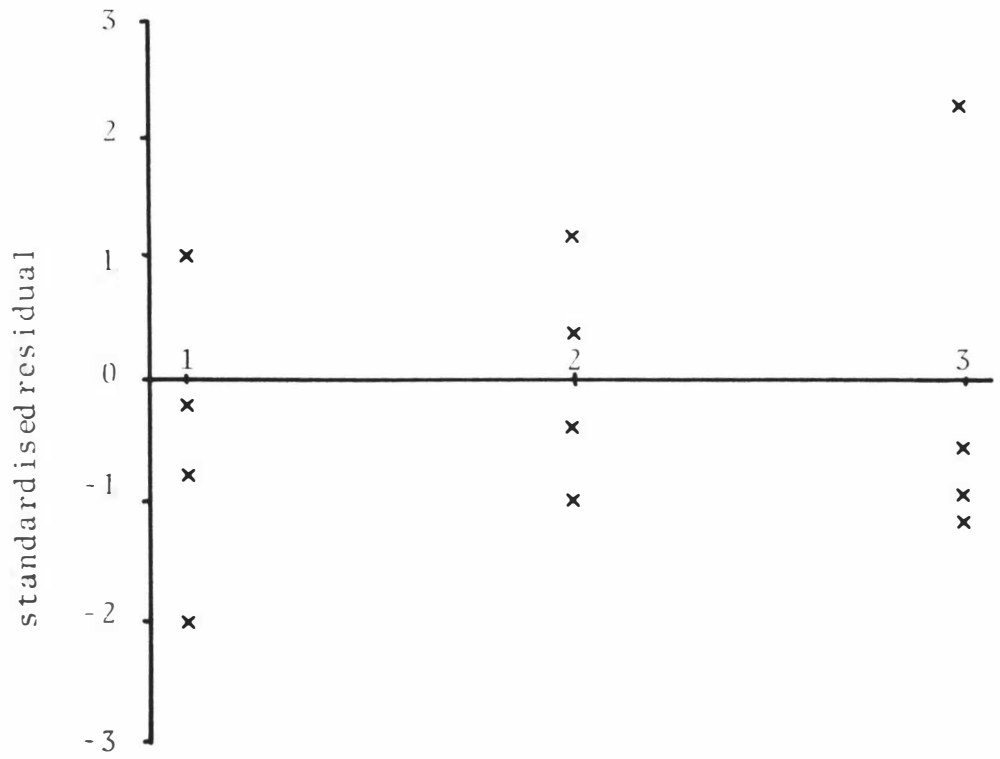


FIGURE D2: Standardized residual and hydraulic retention time