5. Materials and Methods

5.1. BFBR arrangement

The laboratory test BFBR is schematically shown in Figure 1(a). The parts of the BFBR are

- a lower reaction chamber (21) where the wastewater is contacted with anaerobic biomass and where gas generated is collected under pressure
- a filter assembly (30 detailed in Figure 1.b) comprising a buoyant filter media contained in a filter chamber (31)
- a upper chamber (22) where filtered effluent is collected
- a gas siphon assembly (40 detailed in Figure 1.c) between the lower chamber and the upper chamber for periodic discharge of gas from the lower chamber
- a return tube (41) between the lower chamber and the upper chamber

The BFBR is made from two 4 inch diameter glass tubes, with QVF-type flanged ends. The filter holder (internal diameter 2.6 cm, length 43 cm) is made from acrylic tube, and its lower end is capped (33). It is bonded to an acrylic partition plate (23). The plate with the filter holder (31) is clamped in between the two glass tubes, thus forming a lower chamber, (21) and an upper chamber, (22). A gas-liquid-separator, (24), with a peripheral effluent launder was provided at the top of the upper chamber. Biomass and biosolids accumulate in the lower chamber where the reaction proceeds. The upper chamber collects filtered treated effluent, but does not contribute to the active reaction volume as it does not contain biomass. The total volume of the reactor was 11.9 l and
the active volume of the lower feed chamber was 3.5 l. The upper chamber could collect 7.9 l of treated effluent. A gas collection dome was provided on top of the upper chamber.

The filter chamber has holes through which liquid can flow between the upper and lower chambers through the filter bed contained inside. The filter bed, (32), is made from polystyrene balls (porosity 42%, void ratio 0.73, filter bed depth 12.5 cm). It forms a floating granular filter - "Buoyant Filter".

During operation, gas formed due to bioconversion and gas recirculated by pump (3) collect in the lower chamber forcing liquor into the upper chamber through the filter chamber. As a result of filtration action, biosolids and sludge are captured in the buoyant filter. After a predetermined quantity of gas has accumulated in the lower chamber, it is released into the upper chamber automatically by the gas siphon (40) discharge mechanism. Gas release causes a rapid backflow of filtered liquor from the upper chamber to the lower chamber, causing the buoyant filter bed to fluidise and expand downward. Solids captured in the buoyant filter are washed out, "backwashed", into the lower chamber. The interval between successive backwashes is adjusted so as to prevent excessive build-up of filter pressure drop.

The backwash system by automatic gas discharge is shown in Figure 1(c). The system is made from a length of silicon rubber tubing, OD 6mm, ID 4mm, (42) inserted into a 15 mm acrylic tube (41). Acrylic tube 41 called the ‘return tube’ pass through the partition plate (23). The joint between the return tube 41 and the partition plate 23 is sealed against gas leak. The lower end of tube-42 emerges from hole on the side of the return tube and is bend back to form a U as shown in the figure. The return tube extends about 5 cm below the lowest part of the U and is always immersed in the liquor in the lower chamber. The side hole in the return tube is sealed to prevent gas entry into the return tube through gap between return tube and U tube. We call the shorter leg of the U as ‘downcomer’ and the longer leg inside the return tube as ‘riser’. The end of the riser is well above the liquid level in the top chamber of the reactor.

During initial operation of the BFBR, the gas discharge system was occasionally fouled by scum entering the downcomer. This is prevented by adding a scum baffle (43) around the downcomer.
Also during the course of reactor operation, the BFBR was modified with a scum recirculation facility for the lower chamber in order to improve mixing. A scum collection vessel (270 ml) is connected to the lower chamber through a large nozzle. Scum along with mixed liquor, overflows into the vessel during backwashing. The gas vent facility, ensures quick filling of the scum collection vessel. Scum collected in 27 is pumped back into the reactor through nozzle F, using pump 4. The pumping rate is adjusted so that 27 is emptied before the next filling during backwashing. When empty, pump 4 merely functions to circulate gas in lower chamber, providing additional agitation.

The liquor in the lower chamber is mixed by gas recirculation using Pump 3. The pumping rate of gas, can be adjusted to change the interval between backwashing, so as to provide the longest possible filter run before backwashing.

The BFBR was provided with a pH control facility (not shown), through scrubbing carbon dioxide from the recirculated biogas, which automatically adjusts for acidification. No control was provided against alkalification.

The seed innoculum was obtained from a conventional pilot-scale biogas plant treating kitchen waste. The data was collected after a prolonged start-up and acclimatisation period lasting several months. Synthetic dairy effluent (Table 1) was prepared daily by mixing whole milk with tap water and sodium bicarbonate, and trace elements. NaHCO₃ was added to maintain an influent alkalinity between 1000 to 1200 mg/l.
Figure 1. Experimental set-up

(a) 15 litre agitated feed tank; (2) feed pump; (3) gas recirculation pump; (4) scum recirculation pump; (5) water seal; (6) wet gas meter. BFBR reactor assembly: (21) lower chamber; (22) upper chamber; (23) partition plate; (24) gas-liquid-solid separator; (25) inlet for recirculated gas and scum; (27) scum collection chamber; (28) scum collection chamber gas vent; (30) filter module.

Nozzles are denoted as: (F) feed; (E) effluent; (G) gas; (S) scum outlet; (R) gas and scum recirculation inlet.

(b) Filter module assembly: (23) partition plate; (31) filter housing; (32) buoyant filter bed; (33) end cap.

(c) Hydraulic gas release assembly. (41) return tube; (42) U-tube. (42D) downcomer leg of U-tube; (42R) riser leg of U-tube; (43) scum baffle.

Liquid levels: (L1) level in lower chamber; (L2) maximum liquid level in lower chamber; (L3) liquid level inside downcomer limb of U tube; (L4) invert level of U tube. Gas discharge takes place when L3 reaches L4; (L5) liquid level inside gas dome; (L6) liquid level in return tube; (L7) liquid level in riser limb of U-tube.

The difference (L6 - L5) = filter pressure drop.
5.2. Filter design

The principal variables in the design of the filter bed are: Filter media characteristics: size, shape, density, specific gravity; filter bed porosity; filter bed depth, filtration rate, allowable head loss and influent wastewater characteristics.

The filter media particle size is a compromise between filtration efficiency and pressure drop. Particle size should be small for greater filtration efficiency. The particle size has to be large to limit pressure drop. The density of the media materials is also a compromise. Separation of filtered solids from filter media by fluidization is improves if there is greater difference in density of filter media from that of the filtered solids. This implies lower media density. Lower filter media density also leads to better and faster reformation of filter bed after backwash. An addition advantage is that the filter material mass is lower and hence the filter cost is lower for materials sold by weight. On the other hand, filter media backwash velocity required for fluidization is lower if filter media density is high – as close to density of water as possible. Since filter is backwashed with the filtered effluent, filter productivity is improved if media density is higher. With materials such as expanded polystyrene, filter media becomes highly compressible when media density is low and this leads to sharp pressure increase during filtration. Finally there practical and economic considerations on manufacture of the media, with commercially available materials that dictate the choice of filter media. Experiments were conducted with various media and expanded polystyrene EPS beads were chosen for fabrication of the filter bed.

The ability to design filters and to predict their performance is based on 1) Understanding of the variables that control the process and 2) A knowledge of the pertinent filtration mechanisms responsible for the separation of particulate matter from the waste water. The complete filtration process essentially consists of two phases: filtration and back washing.

The end of filter run is reached when the suspended solids in the effluent start to increase (break-through) beyond an acceptable level or a limiting head loss occurs across the filter bed. Ideally both these events should occur at the
same time. Once either of these conditions is reached the filtration phase is terminated and the filter is backwashed to remove the material that has accumulated within the granular filter bed.

5.3. Filter media preparation

Expanded polystyrene beads were prepared as filter media. Expandable polystyrene resins contain a blowing agent which has a boiling point around the melting point of polystyrene. When heated the melted resin expanded because of the vaporisation of the blowing agent. EPS resins in various particle sizes are available from LG Polymers and BASF. These resins are used for manufacture of moulded polystyrene packaging cases, popularly called Styrofoam, which is a proprietary name for the material. The resin is expanded first into beads, packed into moulds and fused by further heating. The reticulated pattern seen on polystyrene packing material shows the boundaries of fused beads.

EPS expanded beads are not commercially available and needed to be prepared from the resin. Several methods of heating the resin for expansion were tried. These included heating in hot air oven, boiling in water, and steaming in a closed stirred vessel. The method of steaming while mixing was easiest for preparing larger quantities of beads and uniform expansion was possible.

5.4. Filter backwash

Filters have to be regularly backwashed to prevent choking. Deep bed filters used in water treatment are backwashed at intervals of several hours. Unlike in raw water filtration, the mixed liquor in an anaerobic reactor has very high suspended solids. Correspondingly, backwash intervals would be very short, (several minutes). The BFBR filter bed is backwashed by fluidization by downward flow of the filtered liquor. The fluidization velocity and bed expansion were measured.

5.4.1. Operation of automatic backwash system

The backwash system operates automatically using a hydraulic gas siphon. Gas produced in the lower chamber and gas recirculated from the gas dome collect below the partition plate. The liquid level in the lower chamber as well as
in the downcomer goes down as gas accumulates. When the liquid level in the downcomer reaches L4, the bottom of the U, gas bubbles out through the riser. Since the internal diameter of the U-tube is only 4mm, gas bubbles in the riser fill the cross-section - a ‘slug’, also called a ‘Taylor bubble’ - which pushes out the liquid column in the riser. The liquid column in the riser, pushed out by the gas slug, falls into the return tube from where it flows back into the lower chamber. Gas held under hydrostatic pressure in the lower chamber flows into the gas collection dome rapidly through the U-tube. As the pressure in lower chamber decreases, filtered liquid in the upper chamber flows back into lower chamber through the filter bed. When the liquor level in the lower chamber reaches level L2, it enters the downcomer and fills the riser re-forming the liquid seal. The following relations may be noted.

- Filter pressure drop = the difference between liquid level inside and outside return tube (L6 – L5). Thus a continuous indication of filter pressure drop can be obtained.
- Backwash volume = (rate of gas production + gas recirculation rate) x backwash interval = liquid volume between L2 and L4, i.e., fixed for a U-tube configuration.
- The filter hydraulic load = gas production rate + gas recirculation rate + feed rate.

5.5. Reactor mixing

In the treatment of high solids effluents, mixing and scum formation are likely issues that can affect the rate of conversion. The experimental BFBR is provided with gas recirculation mixing. The rate of gas circulation is limited - a few bubbles per minutes, released from a 4 mm diameter orifice at the bottom of the reactor liquid pool. The breaking of bubbles generates some turbulence at the surface for breaking of foam or scum.

5.6. Reactor pH control

BFBR liquor pH was controlled by removing carbon dioxide from the gas phase. BFBR is provided with a gas recirculation system for mixing. The recirculation gas was passed through an absorber comprising of a packed bed of glass beads with sodium hydroxide trickled over the bed. The pumping of
sodium hydroxide to the absorber was controlled by a on-off pH controller. The sodium hydroxide concentration is measured to establish the amount of carbon dioxide absorbed.

5.7. Model complex wastewaters for experimentation

Filtration tests were carried out with liquor from a canteen waste biogas plant. The liquor was almost completely bulking with no clear interface forming during 30 minutes of settling time. The sludge contained 13 g SS/l. The liquor was used within the day of collection.

Continuous BFBR operation experiments were conducted with complex wastewater with defined characteristics prepared from components, on a daily basis. The complex wastewaters selected for testing were:

1. Synthetic dairy effluent (milk effluent) prepared from whole milk
2. LCFA effluent prepared from oleic acid effluent

Synthetic dairy effluent (Table 1) was prepared daily by mixing whole milk with tap water and sodium bicarbonate, and trace elements. NaHCO$_3$ was added to maintain an influent alkalinity between 1000 to 1200 mg/l.

<table>
<thead>
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<th>Table 1. Composition of synthetic dairy effluent. 10ml of the trace element stock solution was added per litre of the effluent.</th>
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<td>Pasteurised milk (3% fat, 8.5% non-fat solids)</td>
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<tr>
<td>Tap water</td>
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<td>Sodium bicarbonate</td>
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<td>NiCl2</td>
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Long chain fatty acid effluent was prepared by homogenizing oleic acid with equal moles of sodium hydroxide and making up to required volume. The concentration of LCFA effluent was around 2500 mg COD/l. This effluent was used to study LCFA degradation. Except for milk, all other components of the LCFA effluent are as per Table 1.

5.8. Feed system

There are unique experimental difficulties in laboratory reactor studies with complex wastewater. Typically, prepared slurries have to continuously mixed in feed tanks in order to prevent settling. Peristaltic pumps with narrow bore tubing tend to get choked quickly when pumping slurries. Using larger bore tubing reduces flow velocities and solids settle in tubing. When using fats, the emulsions separate quickly and form a scum layer on top the feed tank. Hence, it is very difficult to achieve uniform composition of feed throughout the day. In the reactor studies carried out, complex wastewater was prepared with diluted milk and starch to simulate sewage, diluted milk to simulate dairy wastewater, and oleic acid with sodium hydroxide to simulate a long-chain fatty acid containing wastewater.

An agitated tank was used to mix the contents of the feed tank, but this was possible only to limited extent.

5.9. Analytical methods

pH in the digester was continuously monitored and regulated with a pH probe (Cole Parmer) and a on-off control system as previously mentioned. The electrode was calibrated daily.

Total biogas production was recorded with a wet-gas flow meter (Insref, India).

Alkalinity and total volatile fatty acids (VFA) concentration in the BFBR were estimated titrimetrically according to Anderson and Yang. Individual volatile fatty acids were measured using by gas chromatography (FISONS 8000 series GC, Shimadzu C-R7A computing integrator, FID detector, 6 ft, 2mm i.d. glass column with Supelco CarbowaxWAW, 0.1% phosphoric acid, carrier gas ultrapure helium 20 ml/min, injector 150°C; detector temperature 175°C, oven isothermal 120°C). The detection level was less than 1 mg/l. Column
resolution was maintained by occasional injection of formic acid. Individual volatile fatty acid analysis was carried out only infrequently.

Total organic carbon (TOC) and inorganic carbon (IC) were monitored using total carbon analyzer, Shimadzu TOC-5000 system with detects combustion product carbon dioxide with NDIR adsorption detector. TOC samples (not filtered) were prepared by dilution of with distilled water and sonication for 15 minutes.

Biogas composition was routinely measured using the TOC analyser, where the IC value is taken as carbon dioxide and (TC-IC) is taken as methane. The TOC 5000 instrument is designed for measurement of carbon in liquid samples. It can be used for gas sample injection, and does not require further calibration, as both carbon dioxide as well as methane are measured in mass of carbon by NDIR absorption of carbon dioxide. Systematic errors are possible in gas sample introduction and these were avoided by calibration with gas standards. Biogas was collected from the reactor in a homemade glass bulb with an acidified water seal. The automatic injector of TOC 5000 has a capillary suction tube made of teflon, which can be inserted into the gas bulb through the water seal. Initially gas composition was verified against gas chromatography measurements (Fisons 8000, TCD, 2mm i.d. silica gel column, He 150 ml/min, oven 40°C, injector 110°C, detector base 120°C, detector wire 190°C) and found to be accurate. The TOC method was chosen over GC because of ease of operation. The TOC method for gas analysis consistently gave less than 1% coefficient of variation in multiple injections.

Chemical Oxygen Demand (COD) tests were carried out according to the open reflux method as per Standard Methods [20] using a temperature controlled block digester (Tecator 2000).

Long Chain Fatty Acids (LCFAs) in the effluent and mixed liquor were determined by gas chromatography after extraction with hexane and esterification of the acids (Fisons 8000 gas chromatograph, Supelco OV1 capillary column (30m X 0.32mm X 0.2), carrier gas ultrapure helium 2ml/min; carrier gas split ratio of 30:1; injector temperature 2500C; detector 3000C; oven temperature program 1500C hold 4 minutes, 100C/min to 2200C hold 2 minutes, 70C/min to 2800C hold 4 minutes). The gas chromatogram was
 calibrated with fatty acid methyl ester (FAME) mix standard, C14 – C22 (Supelco, Bellefonte, PA). FAME mix standards were prepared in hexane, sealed with septum caps in standard bottles and stored at –40°C. LCFA was extracted from mixed liquor as follows: One ml of the sample was withdrawn to a screw-capped vial, 5ml hexane was added and stirred for 30 minutes with a magnetic stirrer 30 minutes. The organic layer was transferred completely to another glass vial and dried off completely by placing the vial in a water bath at 800°C. The dried sample was then cooled to room temperature, 2 ml methylating reagent was added. The vial was tightly capped, sealed and again placed in the water bath for 1 hour for esterification. After methylation, the vials were cooled to room temperature; 2 ml hexane was added and shaken well for 2 minutes. Completeness of esterification was checked using TLC plates. One ml of the esterified sample was pipetted out and capped tightly in a septum lined bottle. Samples were stored in freezer below –40 °C, if not analyzed immediately. LCFA extraction from mixed liquor samples were verified by spiking mixed liquor samples containing no LCFA with oleic acid 200 mg/l. Recovery was 100% +/- 5% and blank samples gave nil reading. This shows that cell wall lipids were not hydrolysed or extracted by the procedure. On the other hand, extraction with solvents such as petroleum ether gave more than 100% recovery.

Ammonia -N was measured using Orion specific ion electrode.

Filter performance testing was carried out in a test filter bed 7cm deep. The filter bed was constructed from two different expanded polystyrene resins, which were expanded to required size by heating. The test liquor was pumped through the bed and pressure development noted by measuring (L6-L5), the rise in liquor level in the return tube.