Journal of Environmental Biology **April 2007,** 28(2) 517-521 (2007)

Treatment of spent wash in anaerobic thermophilic suspended growth reactor (ATSGR)

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(Received: January 10 , 2005 ; Revised received: September 5, 2005 ; Accepted: October 17, 2005)

Abstract: *Pollution through spent wash is a major problem in India. There is an urgent need to develop wastewater treatment technologies for safer disposal. In the present investigation, an attempt has been made to examine a few aspects of thermophilic anaerobic digestion of spent wash collected from a distillery. The study was carried out in a 4 liter laboratory scale anaerobic thermophilic suspended growth reactor. After the successful startup, the organic loading was increased stepwise to assess the performance of the reactor. During the study period, biogas generated was recorded and the maximum gas generated was found to be 11.9 liter at an organic loading rate (OLR) of 29 g COD/l. A 500% increase in the volatile fatty acid (VFA) concentration (1850 mg/l) was observed, when the OLR was increased from 29 to 30g COD/l. During the souring phase the removal of COD, total solids (TS) and volatile solids (VS) were in the order of 52%, 40% and 46% respectively. The methane content in the biogas varied from 65% to 75%.*

Key words: *Thermophilic anaerobic digestion, Spent wash, Biogas*

Introduction

Industrialization generated income has changed drastically the life style of a common man and thereby installation of distilleries in the developing countries such as India, is unavoidable. However the wastewater generated by these industries are complex in nature as they have high amount of organic contents. Approximately 400kl of spent wash or vinasse per annum is generated by over 250 distilleries in India. It is estimated that about 15 liters of spent wash is discharged for every liter of alcohol produced (Kaul *et al*., 1994). The population equivalent of distillery effluent based on BOD load has been reported to be as high as 6.2 billion which means that contribution of distillery waste in India to organic pollution is approximately seven times more than the entire Indian population (Handa and Seth, 1990). Spent wash with a COD of over 1,00,000 mg/l and BOD of 30,000 mg/l ranks high amongst the pollutants produced by industries both in magnitude and strength (Routh and Dhaneswar, 1986). Treatment and safe disposal of spent wash has been a challenge for a long time (Chao, 1983). The high strength of spent wash render aerobic treatment would be too expensive and physicochemical processes have met with little success (Shivayogimath and Ramanujam, 1999).

Thermophilic anaerobic treatment of spent wash is likely to ensure high degree of treatment and recovery of renewable resources. This approach is more attractive for developing and tropical nations such as India, where temperature is suitable for thermophilic anaerobic digestion in most part of the country. Rimkus *et al*. (1982) have reported that the organic matter destruction was comparatively more for thermophilic over mesophilic digestion. Advantages related to the thermophilic digestion include increased pathogen destruction and increased digestion (Rubia *et al*., 2002). In India, variety of anaerobic reactors including anaerobic lagoon (Subba Rao, 1980; Ilyas *et al*., 1998), conventional digester (Parthasarathy *et al*., 1967), anaerobic filter (Gadre and Godbole, 1986) and two staged fixed reactors (Kaul and Badrinath, 1984) have been studied. Literature review reveals that no attempt has been made in India,to evaluate the usefulness of thermophilic anaerobic treatment for treating spent wash and hence the present study has been carried out.

Materials and Methods

Mohan Distillery and Brewery Ltd., Chennai, India has been selected for present investigation and the distillery effluent was collected from the wastewater generated. The samples were kept safe and transported to the laboratory in polyethylene container and stored at 4^0C.

Inoculation: Rumen content of cow was collected from a slaughterhouse, maintaining the anaerobic condition and was used as inoculum. Hobson (1982) has also used rumen content as a source of selection of thermophilic methanogens in their successful investigation. The reactor was seeded with an inoculum of about 30% reactor volume as recommended by Young and Mc Carty (1969) for a suspended growth anaerobic reactor.

Microbial load and composition of the inoculum: The number of microorganisms in the inoculum was counted using hemocytometer and was found to be 35×10^5 cells/ml. The characteristics of the inoculum used are shown in Table 1. Practical difficulties such as pH imbalance and scum formation

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were experienced in selection of thermophilic methanogens from cow rumen content. The selection of thermophilic methanogens from mesophilic flora is a difficult process and only 9% of the flora was capable of growing at thermophilic temperature range (Stronach *et al.*, 1986).

Fabrication and setting up of thermophilic anaerobic digester: The laboratory scale digester has been fabricated and used for the study is shown schematically in Fig.1. A 5 liter capacity, round bottom glass container with a working volume of 4liter (borosil make) was used as the digester. It was placed in the water bath (make:buchi) and the temperature 55±2°C was maintained. The mouth of the reactor was closed with a lid having three openings. Through one of the openings a glass tube was inserted to reach the bottom of the reactor. This served as the outlet for digested waste. Another tube inserted through the second opening served as the inlet for the feed. To facilitate the circulation of feed, a common tube was used to connect the outlet and inlet tubes. In the connecting tube provisions were made to place pH (make: elico, model: LI 120) and temperature probes (make: amadigit, model: ad 20). A peristaltic pump was used for circulating the feed. The third opening served as the outlet for gas. Through this a tube was connected to a wet gas meter (make: ritter, model: TG1) for gas measurement. The entire experimental unit was made airtight by applying a chemical sealing agent (m-seal manufactured by Mahindra and Mahindra Company, Pune, India).

Mode of operation: The ATSGR was operated in a semi continuous mode. The hydraulic retention time (HRT) was fixed and it was 2 days. Once in two days 2 liter of effluent was withdrawn followed by feeding the ATSGR with 2 liter of influent without disturbing the sludge bed.

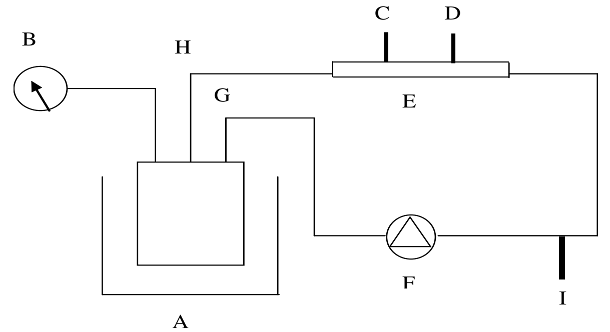
Table - 1: Characteristics of rumen content of the cow

Parameters	Values
Total solids	60,314 ±84 mg/l
Total suspended solids	42,324 ± 53 mg/l
Volatile suspended solids	11,024 ±36 mg/l
Colour	Dark brown

Table - 2: Physicochemical characteristics of distillery effluent

Parameters	Values
pH	4-4.7
Total solids	59000-82000
Volatile solids	38000-66000
Suspended solids	750-1450
COD	82000-11500
TKN	1300-1850
Total phosphate	250-750
Sodium	110-220
Potassium	7000-7200
Calcium	2000-2500
Sulphate	1800-3000

All values except pH are in mg/l



A- Water bath B- Wet gas meter C- pH probe
D- Digital thermometer E- Connecting tube F- Peristaltic pump
G- Sample inlet H- Sample outlet I- Sampling tube

Fig. 1: Laboratory scale anaerobic thermophilic suspended growth reactor.

Chemical analysis: The influent and effluent was analysed for TS, VS, COD and VFA employing methods detailed in standard methods (APHA, 1998). The methane content in the biogas was analysed using a baroda gas chromatograph equipped with a thermal conductivity detector and porapack Q column with hydrogen as carrier gas at a flow rate of 40 ml/min (Srilatha *et al.*, 1995).

Results and Discussion

The distillery effluent was analysed for various physicochemical characteristics and the results are furnished in Table 2. To obtain lower organic load, the distillery effluent was appropriately diluted with distilled water and 1M NaOH was used to correct the pH to near neutral.

Acclimatization phase: The reactor was operated at 55±2°C and started with an initial organic loading rate (OLR) of 2 g/l COD at a HRT of 2 days. This OLR was maintained throughout the acclimatization period and the pH and biogas production during this period were monitored. Fig. 2 illustrates the biogas production and pH variation during acclimatization period. The gas production increased gradually as the phase increased reaching a maximum of 1140 ml at phase 24 (each phase represented a HRT of 2 days). Beyond this phase, the biogas production was stable. The pH of the treated wastewater was in the range of 6.8 to 7.2, which is an indicative of satisfactory reactor performance. The acclimatization was complete after 60 days of reactor operation.

Fig. 3 illustrates COD removal and VFA concentration in the medium during acclimatization phase. The COD removal increased with time and this is in general conformity with the findings of Boopathy and Tilche (1991) during the treatment of industrial wastewater. The COD removal was 31% in the first phase, but gradually increased with increase in phase of operation and reached 67% in phase 21. From phase 21 to phase 30, the reactor showed steady state condition with consistent COD

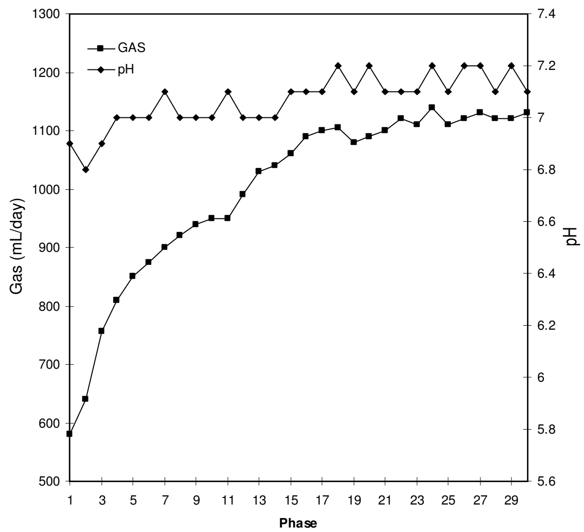


Fig. 2: Biogas production and pH variation in the medium during acclamatization

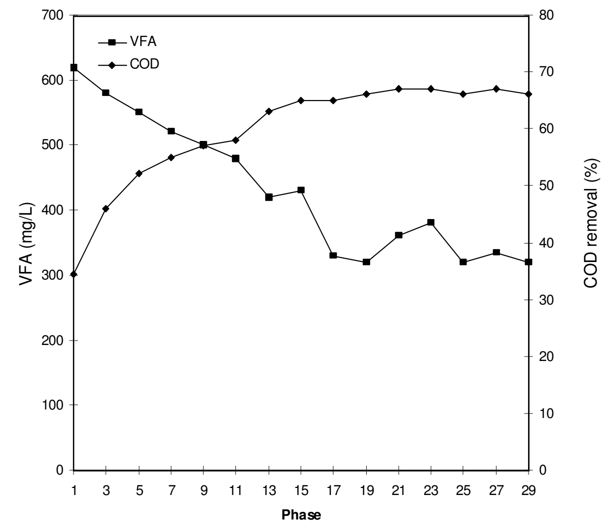


Fig. 3: COD removal and VFA production in the medium during acclamatization

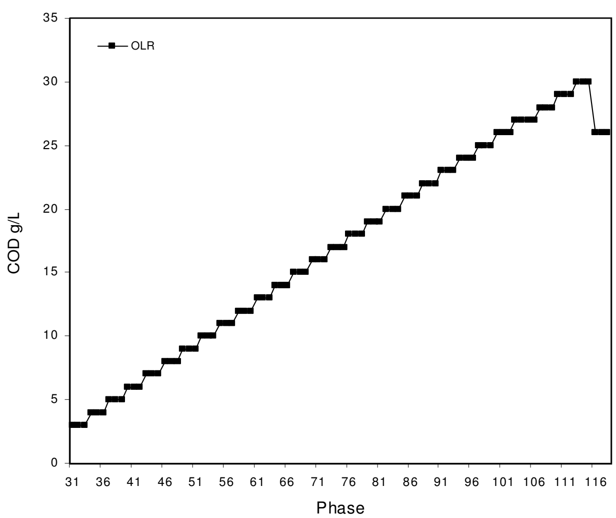


Fig. 4: Loading pattern during the thermophilic anaerobic treatment of spent wash

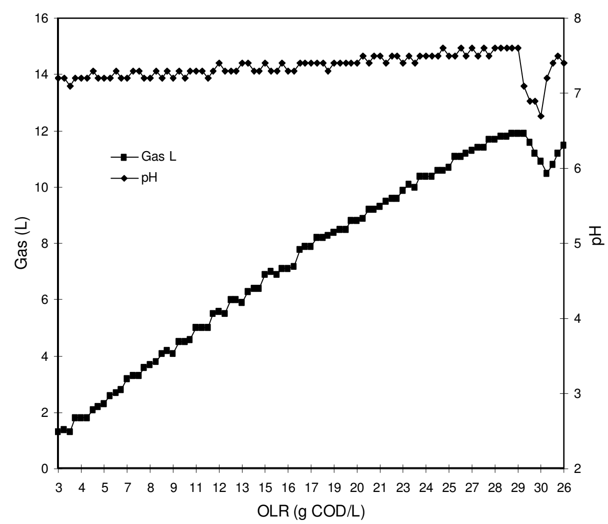


Fig. 5: Influence of OLR on biogas production and pH in the medium during thermpphilic anaerobic treatment of distillery effluent

removal of 67%, indicating successful acclimatization of thermophilic methanogens to the distillery effluent. The VFA in the effluent in phase 1 was 620 mg/l ; it fell down to 500 mg/l on phase 9. Higher levels of VFA in the wastewaters during the initial phases of operation indicate the prevalence of acid fermentation (Van Hanndel and Lettinga, 1994). Subsequently, the VFA in the wastewater decreased and was in the range of 500 to 320 mg/l, indicating healthy anaerobic environment and satisfactory methanogenic activity.

Treatment phase: After successful acclimatization, the reactor was continuously operated for 236 days (including the acclimatization period). Fig. 4 illustrates the loading pattern during the treatment phase. The initial OLR applied during this phase was 2.5 g COD/l. It was increased in a stepped manner to 30 g COD/l over a period of 226 days. The increment of COD between successive OLR was about 0.5 g COD/l.

Fig. 5 illustrates the influence of OLR on pH and gas production during treatment phase. The pH varied from 7.1 to 7.6 up to an OLR of 29 g/l. The pH of the medium dropped to 6.7 when the OLR was increased to 30 g COD/l from 29 g COD/l. It is known that pH value less than 6.8 and greater than 8.3 would cause souring of the reactor during anaerobic digestion

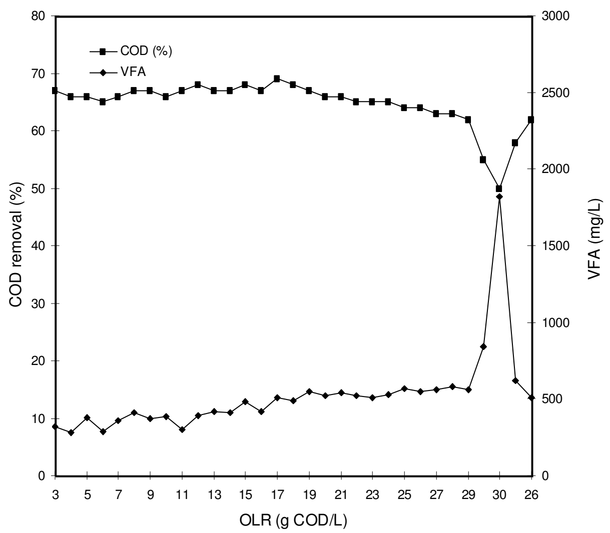


Fig. 6: Influence of OLR on COD removal and VFA production in te medium during thermophilic anaerobic treatment of distillery effluent

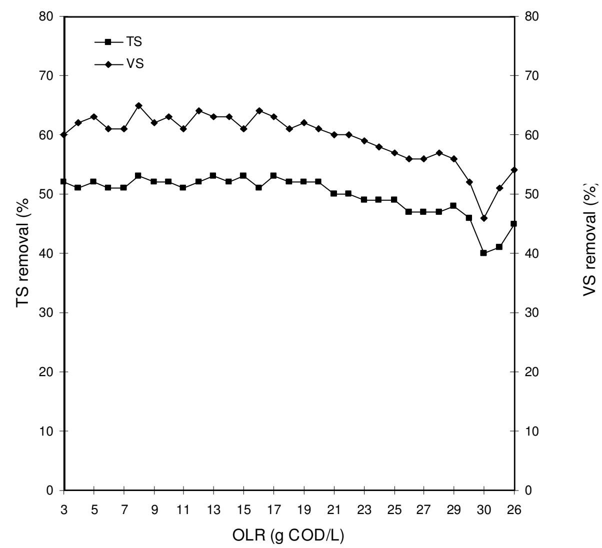


Fig. 7: Influence of OLR on TS and VS removal during thermophic anaerobic treatment of distillery effluent

(Wheatley, 1991). To prevent the souring of the reactor during the present study the OLR was reverted to 24 g COD/l. The reactor started exhibiting signs of recovery and the pH exhibited an increasing trend. The gas production increased as the OLR increased, reaching a maximum 11.9 liter at an OLR of 29 g COD/l. Beyond this loading, the gas production decreased with increase in OLR (30 kg COD/l). The impact of pH decline was reflected in the sigificant decrease of gas production from 11.9 to 11.2 liter when the OLR was increased to 30 g COD/l. The methane content in the biogas varied from 77 to 81%, which is comparable to 75 to 80%, reported during the treatment of spent wash in HUASB (Hybrid upflow anaerobic sludge blanket) reactor by Shivayogimath and Ramanujam (1997).

Influence of OLR on COD removal and VFA concentration in the medium during the treatment of distillery effluent is illustrated in Fig. 6. It is evident from the figure that at an OLR of 3 g COD/l, the COD removal was 67%. As the OLR increased, the COD removal exhibited a gradual increase; at an OLR of 17 g COD/l, the COD removal was 69%. Beyond this, the COD removal was stable and was in the range of 62 to 68 % upto an OLR of 29 g COD/l. The COD removal touched an all time low of 50% when the OLR was increased to 29 g COD/l.

During the stable operational phase (OLR: 3 g COD/l to 29 g COD/l) of the reactor, the VFA (as acetate) levels in the medium varied from 280 to 580 mg/l. VFA started building up in the medium as the digestion proceeded and a maximum concentration of 1850 mg/l was recorded at an OLR of 30 g COD/l. VFA has been recognized as one of the important intermediates during anaerobic digestion (Wang *et al.*, 1999) and is considered a central parameter for anaerobic treatment (Ahring and Angelidaki, 1997). The impact of VFA accumulation was reflected in the marked decrease of COD removal from 62 to 50% when the OLR was increased to 30 g COD/l. Working on distillery effluent using HUASB, Shivayogimath (1999) has reported a VFA concentration over 1500 mg/l at souring point. To prevent the souring of the reactor during the present study, the OLR was reverted to 26 g COD/l. The thermophilic reactor then started exhibiting signs of recovery and as discussed earlier, the COD removal and gas production increased as VFA concentration in the medium decreased.

Fig. 7 depicts the influence of OLR on the removal of TS and VS from the distillery effluent during the treatment phase. The determination of VS is very useful in wastewater treatment plant operation as it offers a rough approximation of the amount of organic matter present in the solid fraction of wastewater (APHA, 1998). Under stable operation conditions (up to an OLR of 29g COD/l), the removal of TS and VS in the wastewater was in the range of 47 to 52 % and 56 to 65%, respectively. Like all other parameters the removal efficiency of both TS and VS decreased drastically when the OLR was increased from 29 to 30 g COD/l, the respective values for TS and VS being 40 and 46%.

From the above discussion it was found that the reactor performed well upto an OLR of 29 g COD/l with 48hr HRT. Further increase in loading deteriorates reactor performance and its effect was seen in reduced removal of COD, TS and VS. From the result obtained, Thermophilic anaerobic treatment is found as a viable option of treating distillery effluent with a considerable production of renewable energy such as methane. Further studies focusing on the microbial dynamics, impacts of environmental factors and engineering aspects would help evaluate the process and its application and pave way for pilot plant experiments.

References

Ahring, B.K. and I. Angelidaki: Monitoring and controlling the biogas process. Proceeding at 8th international conference on anaerobic digestion, Sendai, Japan. IWA Publishing. pp. 40-45 (1997).

APHA: Standard methods for the examination of water and wastewater, 20th Edn. American public health association, Washington, D.C. (1998).

Boopathy, R. and A. Tilche: Anaerobic digestion of high strength molasses wastewater using hybrid anaerobic baffled reactor. *Water Research*, **25**, 785-790 (1991).

Chao, Y.K.: Performance of a two stage methane digester for alcohol silage derived from sugar cane molasses. *Biotechnol. Letters*, **3**, 555-560 (1983).

Gadre, R.V. and S.H. Godbole: Treatment of distillery waste by upflow anaerobic filter. *Indian J. Environ. Hlth.*, **25**, 54-59 (1986).

Handa, B.K. and R. Seth: Waste management in distillery industry. *J. IAEM*, **20**, 44-55 (1990).

Hobson, P.N.: Production of biogas from agricultural wastes. *In*: Advances in agricultural microbiology *(Ed:* Subba Rao). Butterworth Scientific, London. pp. 523-524 (1982).

Ilyas, M., M. Hasnain Isa, I.H. Farooqui, A. Khurseed and R.H. Siddiqui: Treatment of distillery spent was by anaerobic filter - A water conservative approach. *Indian J. Environ. Hlth.*, **40**, 153-159 (1998).

Kaul, S.N. and S.D. Badrinath: Treatment of distillery wastewater by two stage fixed film reactor, IAWPC (India), Technical Annual-XI, **61,** (1984).

Kaul, S.N., T. Nandy and R.K. Trivedy: Pollution control in distilleries. Enviro Media, Karad (1994).

Parthasarathy, T., D. Thangadurai and N. Basheer Ahmed: Anaerobic digestion of distillery waste from sugar industry. *Indian J. Environ. Hlth.*, **9**, 110-117 (1967).

Rimkus, R.R., J.M. Ryan and E.J. Cook: Full scale thermophilic digestion at west southwest sewage treatment works Chicago, Illinois. *J. Water Pollut. Control Federation*, **54**, 1447 (1982).

Routh, R. and R.S. Dhaneswar: Anaerobic treatment of distillery spent wash. *Indian J. Environ. Hlth.*, **29 (2)**, 105-117 (1986).

Rubia, M.A., M. Perez, L.I. Romero and D. Sales: Anaerobic mesophilic and thermophilic municipal sludge digestion. *Chemical and biochemical Eng.,* **16 (3)**, 119-124 (2002).

Shivayogimath, C.B. and T.K. Ramanujam: Granulation during start up of HUASB reactor treating distillery spent wash. Proc. Natl. Seminar on Anaerobic Technologies for Waste Treatment. Chennai, India. pp. 118-123 (1997).

Shivayogimath, C.B. and T.K. Ramanujam: Treatment of distillery spent wash by hybrid UASB reactor. *Bioprocess Eng.*, **21**, 255-259 (1999).

Srilatha, H.R. Krishna Nand, K. Sudhakar Babu and K. Madhukara: Fungal pretreatment of orange processing waste by solid state fermentation for improved production of methane. *Process Biochemistry*, **30 (4)**, 327-331 (1995).

Stronach, S.M., T. Rudd and J.N. Lester: Anaerobic digestion process in industrial wastewater treatment, Springer Verlag, Berlin (1986).

Suba Rao, B.: Distillery effluent treatment, IAWPC, *Technical Annual* - VII, 83 (1980).

Van Hanndel, C. and G. Lettinga: Anaerobic sewage treatment: A practical guide for regions with a hot climate. Wiley publishers, U.K. (1994).

Wang, Q.H., M. Kuninobu and Y. Kato: Degradation of volatile fatty acids in highly efficient anaerobic digestion. *Biomass and Bioenergy*, **16**, 407-416 (1999).

Wheatley, A: Anaerobic digestion: A waste treatment technology. Elsevier Science publishers Ltd. (1991).

Young, J.C. and P.L. Mc Carty: The anaerobic filter for waste treatment. *J. WPCF,* **41**, 160-173 (1969).