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Performance Analysis of Hybrid Electrolysis Enhanced Anaerobic Digestion Reactor in Treating Distillery Spent-wash

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Abstract- In this study performance of HEEAD (Hybrid Electrolysis Enhanced Anaerobic Digestion) reactor was analysed and compared with that of EGSB for treating distillery spent-wash. HEEAD is a new type of reactor, in which, an EGSB (Expanded Granular Sludge Blanket) equipped with electrodes. A direct current (D.C) power supply of voltage of 3.6 V to 28.8 V, used to start electrolysis, resulting in the enhancement of biogas liberation. Two reactors, HEEAD and EGSB were run simultaneously, compared in terms of their performance, stability and biogas production. It has been observed that, reactor stability of HEEAD was better even though COD removal was slightly lesser than that of EGSB and biogas generation had increased up to 73% compared to EGSB.

Keywords- Organic Loading Rate (OLR), Hydraulic Retention Time (HRT), Granulation, Spent-wash, Anaerobic digestion, Biogas.

I. Introduction

The northern part of Karnataka state has a number of sugar industries which produce molasses as a by-product, thus many distilleries have come up very near to sugar industries. Distillery grades as the highest industry amid the list of 17 severely contaminating industries recognised by Ministry of Environment and Forests, Government of India, and are roofed under Central Action Plan [1]. Distillery industries located here make a very severe risk to the environment because of the huge quantity of wastewater generated with a significant amount of recalcitrant compounds leading to broad soil and water pollution [2]. A molasses-based distillery produces 15 L of spent-wash per litre of alcohol produced. Anaerobic digestion is the most appropriate choice for the treatment of high carbon-based wastewaters, since the comparatively low capital costs needed and the possible to produce biogas, a source of energy [3]. This spent-wash is highly acidic, has a very high COD of the order of 1,00,000 to 2,00,000 mg/L and is a threat to the environment if disposed of untreated. The Population equivalent of distillery spent-wash founded on BOD, was as great as 6.2 billion, which means that the input of distillery spent-wash in India to the pollution is about seven times more than that created by the whole population [4]. For the treatment of distillery spent-wash, different physicochemical methods such as precipitation, coagulation, adsorption, and membrane filtration have been applied. But these methods only change but do not degrade the contaminants. In the last decade, the biological methods of treatment gained much importance. The discovery in the anaerobic treatment process resulted in the improvement of high-rate reactors. Anaerobic systems produce a smaller amount sludge than aerobic systems, can switch high strength organic wastes, and unlike most other treatment methods, can result in amplified profits [5]. Microbial degradation and decolourization of industrial wastes is an environmentally friendly and cost competitive alternative of waste minimization. Owing to the different characteristics of wastewater, a different type of treatment methods for distillery spent-wash can be applied by using a combination of different treatment systems [6]. Anaerobic treatment is the most appropriate method for the treatment of wastewater comprising a high concentration of COD since it is cost-effective and environmentally safe [7]. Up-flow Anaerobic Sludge Blanket (UASB), EGSB and fluidized bed reactors are most recognised reactors used in the wastewater treatment. EGSB reactor is a reformed form of UASB reactor categorized through a moderately high ratio of its height to diameter. In the past decade, the EGSB having an up-flow velocity ranging from 4 to 6 m/h became more popular with compact granules along with good settling property [8]. In EGSB, the recirculation keeps diluting the content of the reactor makes it easy to handle the toxic compounds, which UASB cannot [9]. Simate et al.,(2011)[10] treated the distillery waste in a fluidized bed reactor along with Phosphate and Calcium as micronutrients which were disturbing the settling property of sludge. Sharma and Singh (2001)[11], showed that by adding of salts of Nickel, Iron, and Cobalt at 0.1, 10 and 0.5 mg/L respectively to the distillery waste helped a lot in improving its Anaerobic Digestion (AD) by improving both Sludge Volume Index (SVI) and methanogenic activities. The biogas generation potential has been stated by the many authors in the range of 0.15-0.35 m^3 CH_4/kg of COD removed. Partial aeration can improve the methane production in the AD. They observed encouraging response because of oxygenation and also forecasted a best possible oxygenation dose equivalent to a maximum methane yield [12, 13, 14, 15 and 16].

Pirt and Lee (1983) [17] detected that traces of oxygen enriched the AD of algae in the batch reactors. Gerritse et al., (1990) [18] also stated that about 20% growth in methane generation was seen at the small level of oxygen. Botheju et al., (2010) [12] pronounced the usage of partial aeration in AD. Improved hydrolysis and improved acidification in the AD by micro-aeration has been reported [19, 20]. Tang et al., (2004)[21] stated that a micro-aeration of 7.5% (v/v) had

led to oxidation H_2S, no change occurred in the biogas production. The similar results were noticed by Polanco et al., (2009) [22] at partial aeration of the range of 0.013 – 0.024 L O_2/L. d. Micro-aeration in anaerobic reactor permits the organic waste to decompose while reducing the potential for H_2S formation. The effectiveness of partial-aeration for controlling H_2S in biogas has been established by a number of laboratory scale studies. Chemical and biochemical oxidation of H_2S is quick in the AD compared to aerobic digestion [23]. It was stated that about a 20% rise in methane production was at low oxygen concentration in a chemostat in case with headspace oxygen. This was generally due to upsurge of the rate of the hydrolysis of the biodegradable matter and their liquefaction by exoenzymes, generated by the anaerobic digestion, which is mainly aerobic in nature [24].
North Karnataka region usually has the ambient temperature in the range of 27^0C-40^0C, which is the ideal temperature for anaerobic digestion. Industry people are opting for high-rate anaerobic reactors. In this study, micro-aerobic circumstances were set and maintained by water electrolysis. Electrically improved hydrolysis is another acceptable situation. Hydrogenotrophic methanogens grow and multiply quicker compared to acetoclastic methanogens [15]. Botheju and Bakke (2011) [16] have suggested that ‘‘electrolytic aeration’’ may represent an advanced concept for use in the process of aeration as pre-treatment. In this study an electric current was used to introduce water electrolysis, thereby liberating hydrogen and oxygen. The hydrogenotrophic methanogens can develop by using CO_2 and H_2 which will help in the methane production. Thus the performance of HEEAD and EGSB were matched to evaluate the consequence of water electrolysis on the COD removal efficiency and methane generation.

II. METHODS

A. Reactor Setup and Instrumentation

Two laboratory-scale reactors EGSB and HEEAD were fabricated by using acrylic pipe with 100 mm internal diameter of and the working volume of 18 L each. In HEEAD, to create electrolysis process, two zinc plates of size 50 x 220 mm were fixed. These plates were separated by 10 mm and connected to DC power supply. The up-flow velocity of 4 m/h was generated. Sampling points were located at an equal spacing of 220 mm along height. The effluent pipeline was 170 mm measured from the top of the reactor. The effluent tube was linked to the water seal to prevent the leakage of gas through the effluent. The recirculation arrangement along the feed inlet was provided to keep the anticipated up-flow velocities. Miclins peristaltic pumps Model PP 30 EX-2C and PP 60 (India) were utilised for the feeding and recirculation respectively. The wet gas flow meter was connected to through the rubber tube to the gas outlet. Fig 1.shows the schematic diagram of HEEAD reactor.

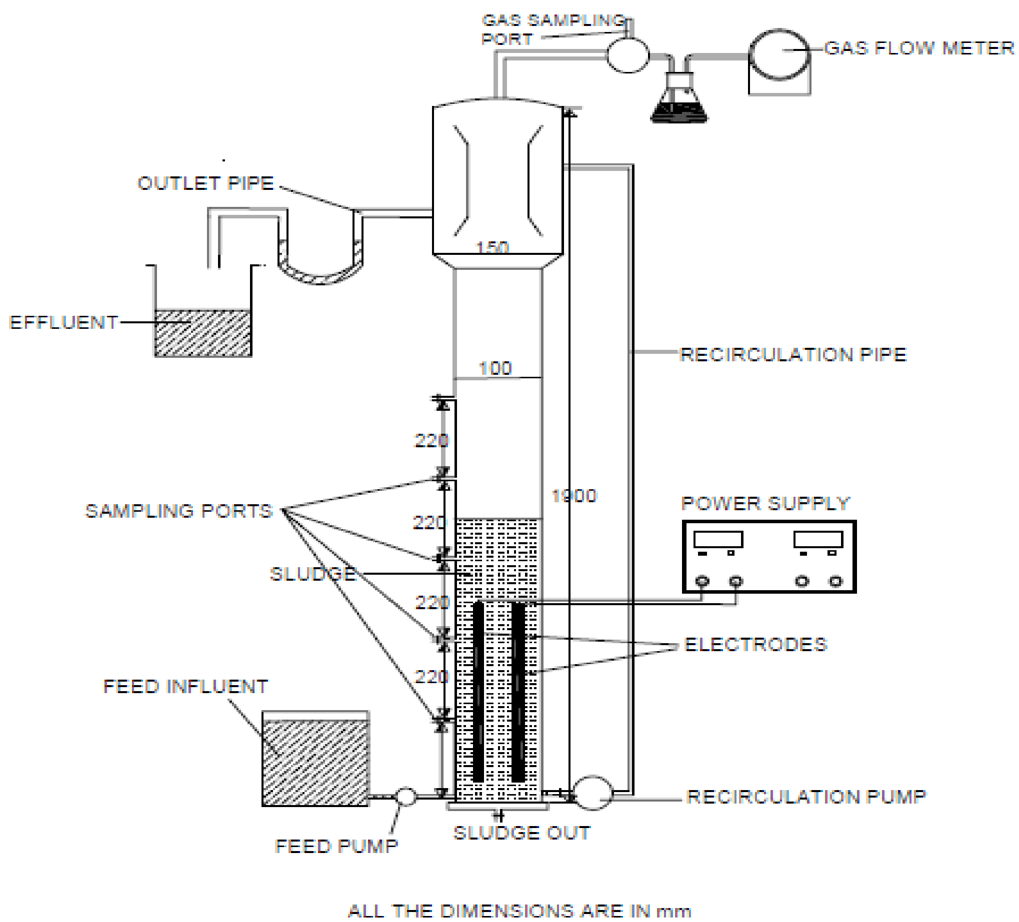

Fig 1 The schematic diagram of HEEAD reactor

B. Inoculum

Inoculum, a non-granulated anaerobic sludge, obtained from Badagandi Sugar Factory, Badagandi, Karnataka, India and was put into batch reactors for 21 days for granulation. The characteristics of sludge used as inoculum charged in the reactor are given in Table I.

TABLE I
CHARACTERISTICS OF ANAEROBIC SLUDGE

Parameter	Value
pH	7.0+/-0.4
Total suspended solids (g/L)	40.6
Volatile suspended solids (g/L)	19.39
Colour	Dark grey
Volume of sludge (L)	9.0
Depth of sludge bed (m)	1.145

C. Substrate

Distillery spent-wash used for the study was brought from The Karthik Agro-Industry, Bagalkot, India. and stored in the deep freezer at 4^0C. The characteristics of Distillery spent-wash are given in Table II.

TABLE III
CHARACTERISTICS OF DIETILLERY SPENT-WASH

Parameter	Values
pH	4.2-4.5
Colour	Dark brown
COD	96400-105200 mg/L
BOD	46000-55000 mg/L
Total solids	86400-93600 mg/L
Chloride	3850-4200 mg/L
Sulphate	3940-4340 mg/L
Nitrogen	3940-4350 mg/L
Phosphorous	780-1040 mg/L
Potassium	7900-8710 L

D. Chemicals and Media Composition

1) Macronutrients

Biological systems have the nutrient requirement, which must be satisfied to a degree equal to the degree of activity wanted. The complete absence of even one of the nutrient can result in termination of biological functions. Often, COD: N: P ratio of 350: 5: 1 was used to describe the nutrient necessity as a function of organic load for the distillery. Nitrogen requirement was based on the practical chemical structure of the microbial cell. Microbial need of phosphorous in AD was around 1/5 to 1/7 of that of nitrogen [25].

2) Micronutrients

The various chemicals required for the EGSB and HEEAD reactor operation contained were methanol, yeast along with basal medium (BM) during the start-up period. BM had all the active micronutrients for growth anaerobic bacteria. The chemical constituents of the BM were as follows $(NH_4)_2.HPO_4$ (80 mg/L), NH_4Cl (800 mg/L), KCl (400 mg/L), $CaCl_2.2H_2O$ (50 mg/L), $CuCl_2.2H_2O$ (0.5 mg/L), $ZnCl_2$ (0.5 mg/L), $CoCl_2.6H_2O$ (10 mg/L), $FeCl_2.4H_2O$ (40 mg/L), $MnCl_2.4H_2O$ (0.5 mg/L), $AlCl_3.6H_2O$ (0.5 mg/L), $NiCl_2.6H_2O$ (0.5 mg/L),$MgSO_4.7H_2O$ (400 mg/L),$Na_2S.9H_2O$(300 mg/L),H_3BO_3(0.5 mg/L), $NaWO_4.2H_2O$ (0.5 mg/L), Na_2SeO_3 (0.5 mg/L), cysteine (10 mg/L), $NaMoO_4.2H_2O$ (0.5 mg/L), KI (10 mg/L), and $NaHCO_3$ (3000 mg/L) [25].

3) Chitosan

It is a normal polysaccharide, the molecular arrangement is similar to the extracellular polymeric substances (ECP). ECP is generally recognised to help anaerobic granulation. Polymeric chains of ECP improve flocculation by connecting bacteria to form microbial centre which is the first step in granulation [26]. The Chitosan was added 2 mg/g of suspended solids [27]. Chitosan in liquid form used to improve sludge granulation. Granule grew up to 2-2.5 mm diameter within a short period of 21 days in batch reactors

4) Methanol

At start-up, methanol was added to help quick granulation. Methanol stimulated the development of methanosarcina bacteria. Addition of methanol was altered for the changed concentration of feed. Initially the methanol was kept 50% of total influent COD (2000 mg/L). Then methanol was reduced to 25%, 12.5% and 0% on days 6^{th}, 9^{th}, and 13^{th}, respectively, by replacing it with the distillery spent-wash [28].

5) Aluminium Sulphate

Aluminium Sulphate added, worked as a flocculent, thus helped in boosting the sludge granulation [26]. In this study, Aluminium Sulphate was kept about 200 mg/L.

III. ANALYTICAL METHODS

Analytical measurements of pH, Alkalinity, Volatile Fatty Acids (VFA), COD were carried out according to the Standard Methods (1998)[29]. pH measurement was done by using Thermo-Orion 420A+pH meter, alkalinity was determined by titration, VFA by distillation followed by titration, COD by open reflux method and biogas/methane was measured through the wet gas flow meter.

IV. RESULTS AND DISCUSSIONS

In the beginning, the distillery spent wash was diluted to 2300 mg/L. The study was carried out completely at ambient temperature, which varied from 29ºC to 35ºC. The pH of the feed was always maintained at 7.0 by adding the appropriate amount of sodium bicarbonate whenever necessary. The macro and micronutrients were also added to the feed regularly throughout the study. The HRT was kept constant as 24 hours and OLR of 2.3 kg COD/m^3/d.

A. pH

pH is an important parameter to be controlled throughout the AD. The methane-producing bacteria have ideal growth in the pH range between 6.6 and 7.6, while stability may be attained in the formation of methane above a broader pH range (6.0 – 8.0). Both the influent and effluent were checked daily for pH and recorded. In the initial stage, the pH ranged between 6.8 to7.4. Stabilization of pH in HEEAD reactor was noticed as it varied from 6.65 to 7.4. Aeration in AD helped in controlling VFA build up and thus checked pH drop. According to Zitomer and Shrout (1998)[30] in un-aerated reactors, observed a dropping pH faster than partially aerated complete-mixed digesters. Guiot et al., (1999) [31], noticed CO_2 stripping action because of aeration.

B. Granulation

Tay et al., (2006) [32] studied bio-granulation methods to treat different wastewater. To form granules in an anaerobic reactor more number of conditions has to be satisfied. Until now, no model is able to tell the entire anaerobic granulation process in a correct manner. Batch reactors were run to investigate sludge granulation. Figure 2. shows the granulation on 21stday. After careful observations it was found that on the 21st day, the average diameter of granules was found to be between 2.0 to 2.5 mm, indicating the successful granulation. This granular sludge was then fed to the HEEAD and EGSB reactors of about 50% of reactor volume and also with a variable feed concentration 2,300 mg/L to 14,200 mg/L. The COD removal efficiency, VFA, and alkalinity were measured every day.

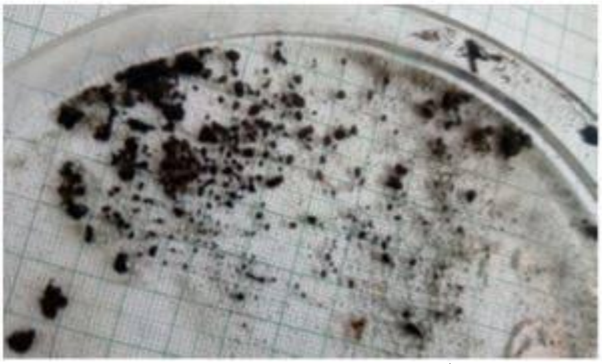

Fig 2 Granules observed on 21st day

C. HRT and Feed Concentration

For both the reactors, the first feed concentration was kept about 2,300 mg/L, which was then extended up to 14,200 mg/L. The HRT varied from 24 to 12 hours for both the reactors. The feed concentration was incremented by 2,300 mg/L whenever the satisfactory COD removal efficiency (>80%) was noticed.

D. Influent COD and Effluent COD

1) EGSB Reactor

Counting the days from the beginning of batch reactor for granulation, EGSB reactor was started on the 23rd day, with COD 2300 mg/L, HRT as 24 hours, at an OLR of 2.3 kg COD/m^3/day. On the 32nd day, the COD removal efficiency was 89.1%. Then COD was incremented to 4,600 mg/L. It was noticed that on the 43rd day, COD removal efficiency reached 85.25%. On 106th day at COD of 14,200 mg/L, at HRT of 12 hours COD removal efficiency touched 94.1%.

2) *HEEAD Reactor*

HEEAD was started on the same day (23rd day) at an OLR of 2.3 kg COD/m^3/day. But as the day progressed, on the 32nd day COD removal efficiency reached 91.6% due to acclimatization. Whenever the reactor reached steady-state, the COD was incremented regularly by an amount of 2,300 mg/L. Figure 3.shows the COD removal efficiency against OLR. On the 43rd day, COD removal efficiency extended to 91.85%. Likewise, it continued, on 106th day at COD of 14,200mg/L, at HRT of 12 hours, it was noticed that COD removal efficiency reached the peak of 96.45%, the methane produced was of 69.76 L.

Using a linear regression equation, H_2 generation was related with applied current:

$QH_2 = KH_2 * I$ ....(1)

where QH_2 is the H_2 flow per day (L d^{-1}), KH_2 is the regression coefficient, calculated by the method of least squares and "I " is the applied current in amperes. In the abiotic test carried before to the start-up and the regression coefficient KH_2 = 12.0 [Eq. (1) was found R^2 = 0.99, with help of 7 measurements]. Applied voltage varied from 3.6 to 28.8 volts and simultaneously the current ranges from 0.17 to 1.76 amperes. Energy spent for hydrogen generation was reasonably high and ranged between 3.38 to 59.67 Wh/LH_2, based on the applied voltage. Once the reactors had reached a steady-state, the HRT was reduced from 24 to 18, 15, and 12 h, along with changing the influent concentration of spent-wash.

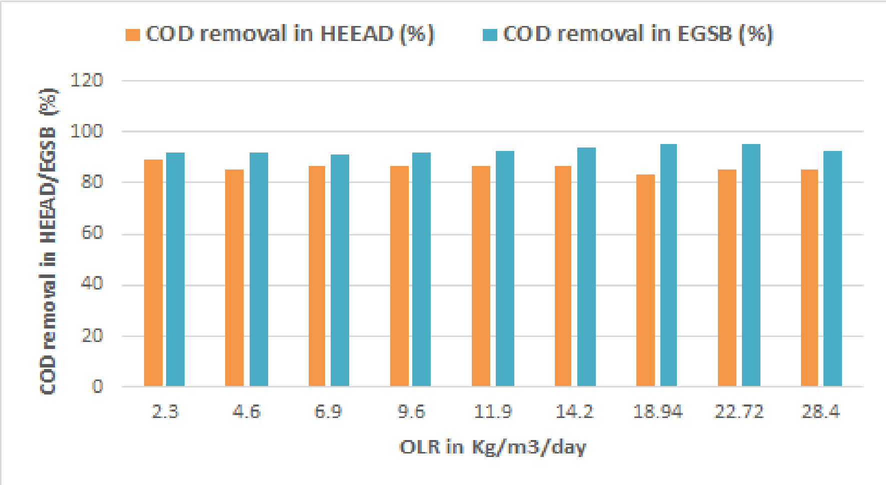

Fig 3. COD removal efficiency V/s OLR

Polanco et al. (2009) [22], observed that an aeration of the range of 0.013 – 0.024 LO_2/L.d did not disturb the biogas production, on the other hand, merely the H_2S oxidation was observed. By reducing the sulfides levels led to the process by lessening the toxicity of sulfides on the methanogenic and acetogenic bacteria and also eradicating the creation of odorous and corrosive gas the H_2S. In our study electrolysis process was started by maintaining the electrode voltage of 3.6, 7.2, 10.8, 14.4, 18, 21.6 25.2 and 28.8V at an OLR of 2.3, 4.6, 6.9, 9.6, 11.9, 14.2, 18.94, 22.72 and 28.4 Kg/m^3/day respectively. Figure 4.shows Oxygen Required/ Liberated against OLR in HEEAD. Here O_2 required was assumed to be O_2 utilised for oxidation of H_2S. At 4.6 and 18.94 OLRs oxygen liberated by electrolysis was equal to the required oxygen, hence there is no chance of the presence of H_2S in HEEAD as all H_2S was completely oxidised to sulphate. At higher OLRs the significant amount of H_2S remained in the reactor, thus was toxic and created a methane reducing activity of acetoclastic methanogens. Electrolysis process released O_2 of the order of 0.04 to 0.53 L O_2/L_R/d. Along with the oxidation of H_2S, hydrolysis/acidification resulted in the reactor as O_2 released was more than in the study by Ponalco et al.,(2009)[22], Tango and Ghaly (1999)[33] and Ghaly and El-Tawwel (1995)[34]. Aeration reduces the lag period of the micro-organisms. However, Conklin et al., (2007) [35], established that partial-aeration led to reducing the digester standby capacity (capacity to bear sudden loads) in an AD, may be limited from the slower action of methanogens in presence of oxygen. The increase in the hydrolysis with partial-aeration was only detected in proteins and carbohydrates, but it was not true in lipids. Figure 5.shows CO_2 Consumed/CO_2 Produced V/s OLR. CO_2 consumed was calculated stoichiometrically by the presence of ‘‘H_2 produced’’ thinking that all H_2 generated was used up. "CO_2 consumed" was almost half of the "CO_2 produced" at high OLRs and the part of the CO_2 got converted to methane in presence of hydrogenotrophic methenogens. The presence of CO_2 in the biogas observed in percentage, converted to litre and plotted ("CO_2 produced"). By micro-aeration, hydrolysis, and acetogenisis was speeded up making the acetoclastic methenogens to grow faster. Thus the overall increase in methenogenic activity was seen in the reactor as methane production. Still, there is ample scope for methane production as “CO_2 produced’’ at high OLR was more.

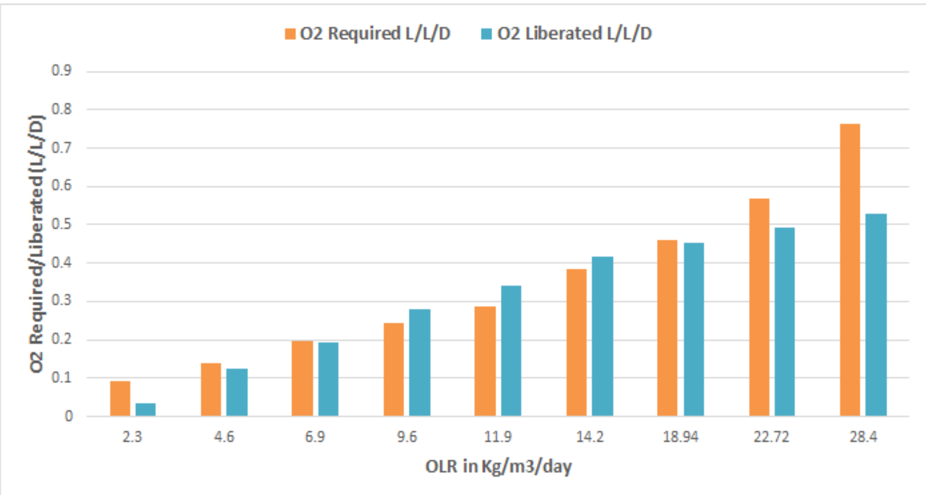

Fig 4. Oxygen Required/ Liberated V/s OLR in HEEAD.

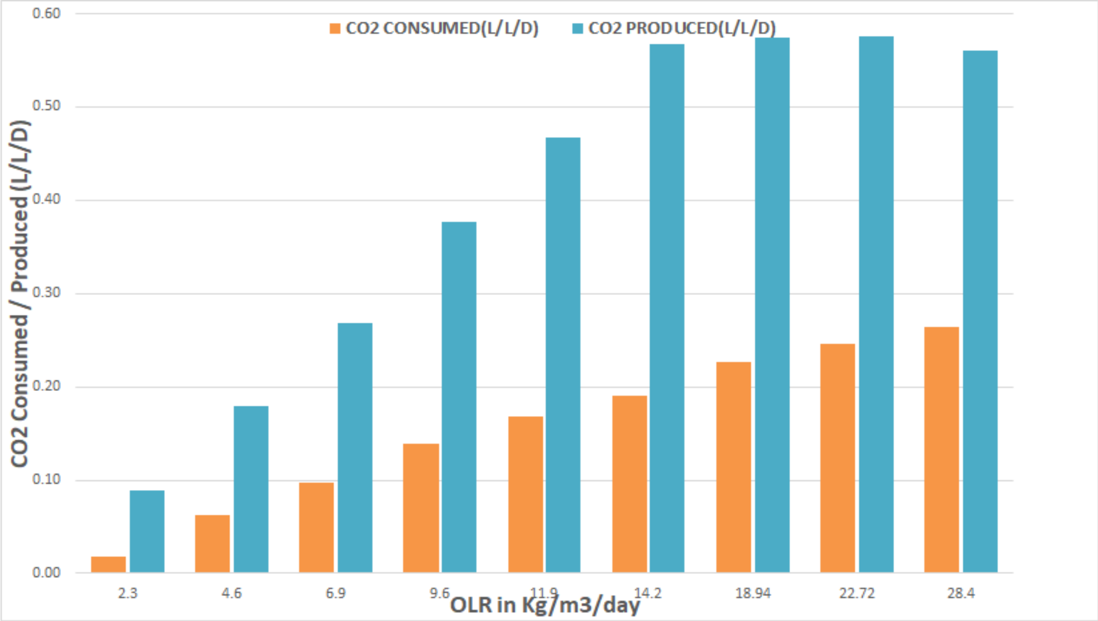

Fig 5. CO_2 Consumed/ Produced V/s OLR in HEEAD

E. VFA, Alkalinity and pH

Enhanced hydrolysis in anaerobic digestion by micro-aeration has, however, been reported by Johansen and Bakke (2006) [19] and enhanced acidification (VFA production) under mild aeration had also been suggested by Chu et al., (1994)[20]. These effects are known to be mostly due to the greater solubilisation (hydrolysis/acidogenesis) of particulate organic matter in the presence of a little amount of oxygen. Aeration can have both positive and negative impacts on biogas production. [36].The aim of this study is to recognize and describe the different effects of aeration on AD. The concentration of VFA is one of the major regulatory parameters in an anaerobic treatment. When COD of feed was increased, it was followed by an increase in VFA levels. Inhibition of methanogenesis can occur at high VFA concentrations. The ratio of VFA and alkalinity is one of the key parameters that influence the reactor performance and COD removal efficiency. In circumstances of overloading the reactors and in the occurrence of inhibitors, the methanogenic activity cannot remove volatile organic acids as fast as it was produced. This leads to the accumulation of acids and the reduction of pH to levels that also delay the hydrolysis. As a consequence, the acids do not build-up beyond the counterbalancing capacity of the alkalinity in the medium, thus pH remains in a range which helps in growth of the methanogenic bacteria [25]. An increase in loading was followed by increase in the VFA levels. However as biomass got acclimatised, the COD removal efficiency increased and VFA levels also were reduced. Even though VFA concentrations were varying the pH of effluent, pH varied between 6.9 to7.6 in EGSB whereas pH varied between 6.0 to7.5 in HEEAD, showed the existence of adequate buffering capacity in the system. This buffering capacity can be attributed to neutralization of the feed by sodium bicarbonate.

F. VFA-Alkalinity ratio with varying OLR

If the ratio of VFA / alkalinity more than 0.71, the inhibition of methanogens occurs and the temporary process fails, increase above 0.3 to 0.4 shows system uncertainty and a proper ratio is to be preserved between 0.1to 0.2 [25]. Figure 6.shows the VFA / Alkalinity ratio against OLR. The decrease in VFA in the wastewater indicates a healthy anaerobic environment and satisfactory methanogenic activity. The variations of VFA and alkalinity during the study period, ratio showed a continuously decreasing trend i.e 0.22 to 0.18. and 0.18 to 0.14 in the HEEAD and EGSB reactors respectively. The VFA/ Alkalinity ratio was a little bit higher in HEEAD, but in no case, it had never crossed 0.3.

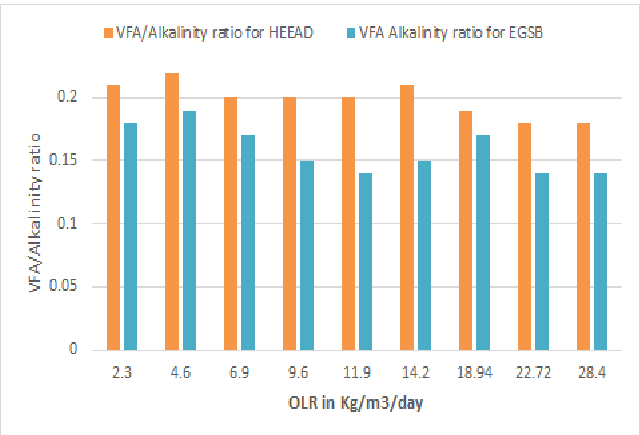

Fig 6.VFA / Alkalinity ratio v/s OLR

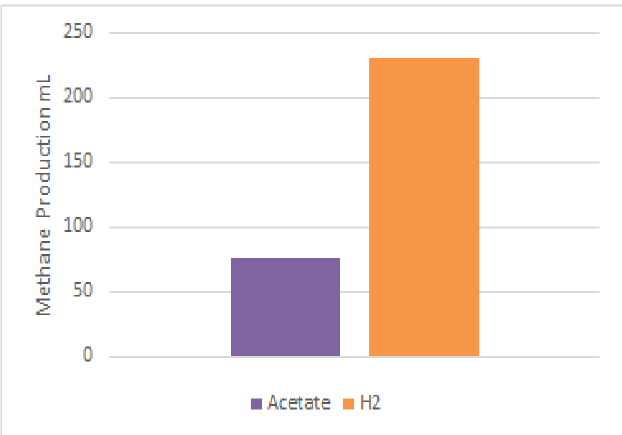

Fig 7. Methane production observed in batch activity tests

G. Methane Production and Methane Yield

1) Methane production

Both tests were carried out in 500 ml bottles. The first bottle was fed with sodium acetate (2g COD/L) and volatile solids (2.5gVS/L) which were then filled with gas CO_2/N_2 (20%/80%) and afterwards incubated at 35 ^{0}C in a rotary shaker at speed of 400 rpm. The second bottle was fed with distillery spent-wash (2g COD/L) and volatile solids (2.5g/L).Figure 7. Shows the methane generation rate observed in the batch study. In hydrogen consumption tests, the bottle headspace was filled with CO_2/ H_2 (20%/80%), under pressure of 10–15 kPa, and incubated at 35 ^{0}C in a rotary shaker, at speed of 400 rpm (Carlos, 2007)[25]. Methane production in the hydrogen-filled bottle is almost three times greater than that obtained with nitrogen. This behaviour states that there is still large scope for formation of methane, as shown in Figure 8.

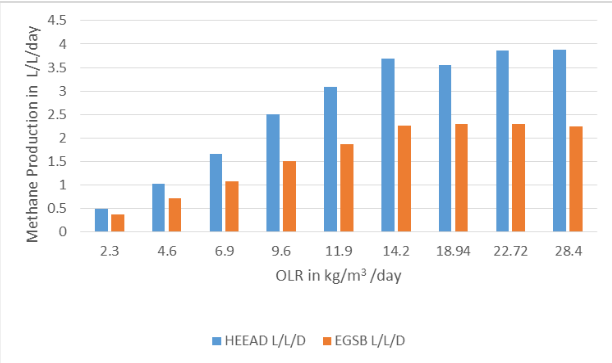

Fig 8. Methane Production related to OLR

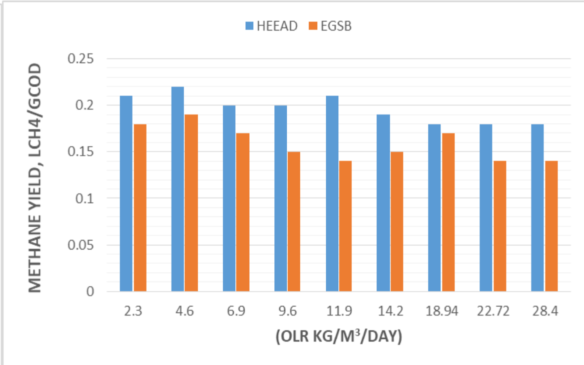

Fig 9. Methane Yield at different OLRs

At an OLR of 2.3 and 28.4kg COD/m^3/day, HEEAD showed methane production of 0.492 and 3.876 L/L/day respectively whereas EGSB showed methane production of 0.36 and 2.24 L/L/day respectively. It was observed that an increase in OLR had a positive result on the methane production in both reactors. At different HRTs (OLRs) the two reactors were run and production of methane was noted.

2) Methane yield

It is documented that partial aeration can improve the methane yield in the AD [Gerritse et al. 1990] [18]. Gerritse et al., (1993)[37], quantified that about 20% rise in methane generation was seen at small oxygen levels in the chemostat (dilution rate 0.06 h) on condition that with headspace oxygen. The methane yield was calculated during the steady-state condition of the reactor using the formula shown below.

YCH_4 = Methane flow rate (L/d) / Liquid Flow rate (L/d) (COD influent - COD effluent)[38].

Figre 9. Shows Methane Yield at different OLRs Methane yield showed an increasing trend with increasing OLR and became stable from OLR of 9.6 Kg/m^3/day. Maximum methane yield of 0.3 was observed at OLRs of 9.6, 11.9 and 14.2 Kg/m^3/day. At OLRs 18.94, 22.72 and 28.4 both the reactors showed a decreasing trend.

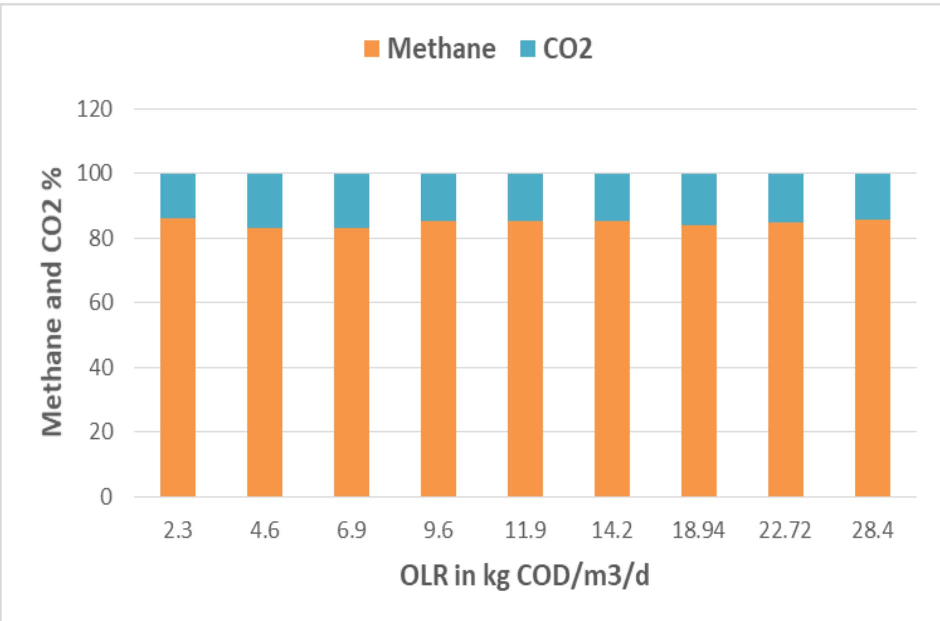

Fig 10. Methane and CO_2 in percentage V/s OLR in HEEAD

V. CONCLUSIONS

The two laboratory-scale HEEAD and EGSB reactor were compared for the treatment of distillery spent- wash for different OLRs for a duration of 106 days under mesophilic condition. By using non-granular anaerobic sludge as a seed along with chitosan, the positive effect was noticed on granulation within a short period of 21 days in batch reactors. Addition of chitosan helped in the formation of larger granules with higher methane production and higher COD removal efficiency. HEEAD reactor showed COD removal efficiency of 85.3 % for OLR of 28.4Kg COD/m^3.day with HRT of 12 hours but for the same OLR in EGSB, COD removal efficiency was 96.45%. Water electrolysis was helped to stabilize the pH within HEEAD. Due to electrolytic-aeration, oxidation of H_2S was noticed along with faster hydrolysis and acidogenisis. Hydrogenetrophic methanogens grew faster in presence of H_2 and CO_2 forming methane. Thus methane production in HEEAD reactor was 1.73 times higher than that of EGSB reactor.

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