

An experimental study on biogas production by anaerobic digestion of rice mill wastewater

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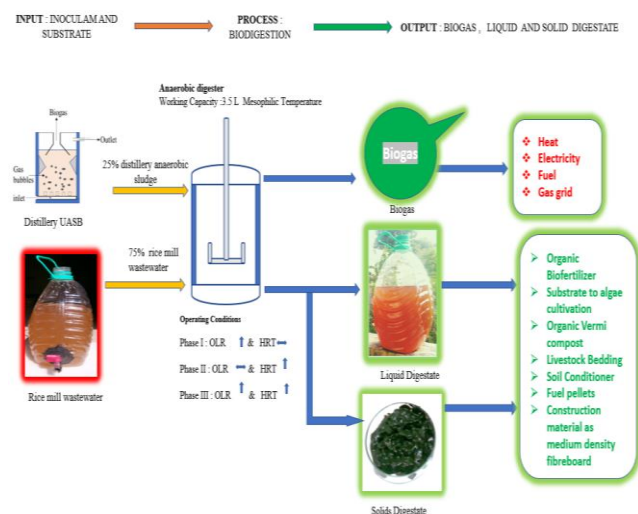
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Graphical abstract



Abstract

With the rising interest for renewable power source and ecological security, anaerobic digestion of biogas technology has attracted a considerable attention among researchers. This study proposed a new technique for producing biogas from rice mill wastewater (RMW) by utilizing an anaerobic digester. An anaerobic digester was packed with RMW and distillery anaerobic sludge at a mesophilic temperature for 15 days for stabilization. After that the effect of the organic loading rate (OLR) and hydraulic retention time (HRT) on the mesophilic anaerobic digestion of RMW was examined. The OLR of the anaerobic reactor was increased stepwise from 0.25 to 3.91 kg COD/m³/day, and the HRT was varied from 1 to 32 days. The total chemical oxygen demand utilized was >75%, and the CH₄ percentage of the biogas was 62%–63% for the OLRs studied. The efficient working volume of the digester was maintained as 25% of the distillery anaerobic sludge and 75% of RMW loaded at a mesophilic temperature for the study purpose. By changing the conditions of the OLR and HRT, biogas production, methane yield, and percentage of chemical oxygen demand (COD) reduction were examined. This enhanced the biological treatment of

the effluent with the anaerobic sludge in a continuous activity mode. The observed results showed that the proposed technique exhibited in prominent biogas production with maximum COD reduction when compared with existing approaches.

Keywords: Rice mill wastewater, anaerobic digester, mesophilic condition, organic loading rate, hydraulic retention time, biogas production, chemical oxygen demand reduction, methane yield.

1. Introduction

Owing to rapid industrialization, natural debasement has been increasingly used as a noteworthy test worldwide (Nielfa *et al.*, 2015). Understanding how a clean environment is fundamental for the smooth living and better wellbeing of people has become crucial (Beyene *et al.*, 2014). Anaerobic treatment of organic effluents has two benefits: prevention of contamination and production of energy as biogas (Tsado, Adamu and Owolabi). Globally, rice is an important nutritious staple for a large portion of the general population, and 90% of rice is grown and consumed in Asia. Of the total proportion of rice grown in the world, approximately 22% was contributed by India in 2011–2012 (Veluchamy and Kalamdhad, 2017). Rice mills in India vary broadly in their abilities. Some rice mills are huge with motorized offices and contamination control gadgets; however, the majority of small rice mills face a shortage of these gadgets (Malamis *et al.*, 2013). All the rice mill effluents originate from the parboiling of rice (Kamali and Khodaparast, 2015; Noguchi *et al.*, 2017). Rice mills release wastewater, particulate matter, and solid wastes. Poor structure or treatment systems of rice mills leads to ineffective treatment of wastewater containing an abnormal concentration of toxins (Singh *et al.*, 2018).

Transfer of this wastewater into water bodies can be lethal to oceanic life. Various studies have focused on the treatment of rice industry wastewater (Xin *et al.*, 2018). However, the wastewater released after treating it with various purification techniques currently remains harmful because it contains a significant number of serious issues (Dhanasekar and Sasivarman, 2016; Nain *et al.*, 2015). Therefore, there is a critical need to develop new high-

performance and cost-effective techniques that can efficiently treat rice mill wastewater (RMW) (Hu *et al.*, 2018; Yuan *et al.*, 2012). Anaerobic assimilation is considered a reasonable technique for treating RMW. A recent study reported anaerobic digestion (AD) as an ecologically stable biological treatment process (Liu *et al.*, 2018). AD treatment process has several advantages over other wastewater treatment strategies (Mao *et al.*, 2015). For instance, AD limits the utilization of extensive land regions, does not produce foul smell, and reduces organic load and pathogens while producing methane and organic manure as the final metabolic products (Haider *et al.*; Weng *et al.*, 2014). Compared with other techniques used for treating RMW, AD is more cost effective because it yields methane and slurry as the final products that can be used as an energy source and organic compost, respectively (Leju *et al.*, 2012). In an anaerobic digester system, biogas can be produced from different organic wastes and thus can be used as a critical sustainable power source, resulting in a low carbon footprint.

In 2018 Lu, D., *et al.* (Lu *et al.*, 2018) explored the viability of utilizing biogas generated from the AD of septic tank sludge and its anaerobic center for microalgal development. Three types of sparging gas (biogas, air, and nitrogen gas) and two culture media (anaerobic center and bold basal medium) were utilized to generate six different test settings. The highest microalgal development of 1074 mg volatile suspended solid (VSS)/L was obtained when both biogas and the anaerobic center were utilized following 10 days of digestion at 30 °C.

In 2018 Xu, R. (Xu *et al.*, 2018), determined the progression of AD-related microbiome facilitating assorted individuals from the phylum Actinobacteria, Bacteroidetes, Euryarchaeota, and Firmicutes, which were affected by the organic loading rate (OLR) and hydraulic retention time (HRT). The OLR resulted in better microbial system modules than did the HRT, indicating the further subdivision of useful parts. The strongest pairwise link between firmicutes and biogas production demonstrated that microbe's dependent on the system can be considered as markers of AD.

In 2014 (Okeh *et al.*, 2014) explored lab-scale biogas production from rice husks generated from various rice mills by using cow rumen liquid as the source of inoculum. The effects of different parameters, namely water dilution, starting pH, overwhelming metals, and nitrogen sources, on AD were assessed. The highest biogas generation rate of 30 and 69 mL/day was obtained for the control and poultry droppings, respectively, following two days of digestion, whereas urea produced 8 mL/day of biogas on day four of digestion.

Zealand examined the generation of methane (CH₄)-rich biogas (45%–55%) from waste rice straw (RS) through AD as an option in contrast to consuming while being reasonable. Five 2-L reactors were utilized to examine the effect of the feeding frequency (FF) and OLR on the anaerobic assimilation of rice straw with inflow rates between five doses every week and one dose every three weeks. The best biogas yield was 300mL/L of reactor/d at

half CH₄ at the lowest OLR1, and the mean yield of biogas was 447mL/L/d at 52%CH₄ under the most favourable reactor conditions.

In 2018, Feng (Feng *et al.*, 2018) explored the bio electrochemical anaerobic digestion of sewage sludge with an HRT of 10 days and compared it with that at a mesophilic condition (35°C). For an HRT of 20 days, the bio electrochemical anaerobic assimilation at an ambient temperature was found to be more stable. Bio electrochemical anaerobic absorption that can spare the thermal energy input at an ambient temperature is recommended for the treatment of organic waste incorporating sewage sludge in regions with a moderate and cool atmosphere.

In 2015, Giri and Satyanarayan (Giri and Satyanarayan, 2015) proposed the treatment of wastewater resulting from the parboiling of rice by utilizing an anaerobic settled film bed reactor. Four organic loadings of 0.8, 1.6, 2.4, and 3.2 kg chemical oxygen demand (COD)/m³/day were examined. Results demonstrated that the biochemical oxygen demand (BOD) and COD reduction ranged from 83.0% to 92.7% and 80.2% to 89.0%, respectively, when Biopac media were used in the reactor system and from 79.4% to 90.6% and 76.7% to 86.1% respectively, when fugino spiral media were used. The results indicated that the Biopac media-packed fixed- film reactor was more efficient at treating parboiled rice manufacturing wastewater than the Fugino spiral media-packed reactor.

In 2016, Saini (Saini *et al.*, 2016) treated RMW by using an upflow anaerobic sludge blanket reactor. The experiment was divided into three stages: Stage I, II, and III that were operated at 50%, 100%, and 100% RMW feed at an HRT of 20 h, 14 h, and 10 h, respectively. Ideal operating conditions resulted in an 86% reduction of COD even at a lower HRT of 10 h and a higher OLR. The results indicate that the OLR affects the COD reduction effectiveness of the reactor.

Rice production requires a substantial amount of water for soaking of the paddy. The amount of water required for the soaking process is generally 1.3 times the weight of paddy, and approximately 8 tons of paddies are soaked in a typical rice mill per day, which makes this process the major contributor to wastewater discharge. The wastewater released from the parboiling process that has a high BOD and COD constitutes the major source of water pollution. Therefore, there is a need to develop new efficient and cost-effective technologies for treating RMW.

Objective of this study used the varying conditions of the OLR and HRT (phase I: keeping the HRT constant and varying the OLR, phase II: keeping the OLR constant and varying HRT, and phase III: varying both the OLR and HRT) to determine the most optimum conditions for biogas and methane production as well as a reduction in COD.

2. Material and methods

2.1. Chemicals

All reagents and chemicals were purchased from Merck Life Science Private Limited, Mumbai, India. All reagents,

chemicals used were in analytical grade and utilized as such without any additional purification. Double distilled water was used for analysis and experiment's purpose.

2.2. Experimental investigation of RMW treatment

In the proposed experimental analysis, biogas was produced from the RMW industry. Normally, wastewater coming from rice mill operations containing a high concentration of organic and inorganic substances cause significant pollution. AD is considered a suitable method for treating RMW. The anaerobic digester was loaded with RMW along with distillery sludge at a mesophilic temperature. The conditions of the system stabilized after 15 days of digestion. A continuous stirred tank reactor with a working capacity of 3.5-L was filled with 25% distillery anaerobic sludge and 75% RMW, and the produced biogas was monitored on daily basis (Figure 1).

2.3. Characterization of materials

Inoculum: For the experimental analysis, the reactor was inoculated with the effluent and anaerobic sludge; that is, paddy soaking wastewater collected from M/s Muthuraja, Modern rice mill industry, Pudukkottai, Sivaganga district, Tamil Nadu, and sugar distillery anaerobic sludge collected from EID Parry (I) Ltd., Nellikuppam, Tamil Nadu. In the anaerobic digester, was as follows: 2.625 L (75%) of RMW and 0.875 L (25%) of distillery anaerobic sludge were added. After determining the physicochemical properties, the wastewater was put away in a fixed holder and kept in a cold room at 4°C for further examination and analysis. (Table 1)

Table 1. Characteristics of rice mill wastewater

| Parameters | Units | Value |
|-----------------------------------|-------|-------------|
| Color | - | Pale yellow |
| pH | - | 4.51–5.10 |
| Total alkalinity | mg/L | 500–750 |
| Total dissolved solids(TDS) | mg/L | 2030–2460 |
| Total suspended solids(TSS) | mg/L | 850–1170 |
| Total solids (TS) | mg/L | 2880–3630 |
| BOD | mg/L | 2550–2950 |
| COD | mg/L | 4250–5120 |
| Volatile suspended solids (VSS) | mg/L | 1200–1400 |
| Volatile fatty acids (VFA) | mg/L | 790–910 |
| Total kjeldahlnitrogen (TKN) as N | mg/L | 32–44 |
| Calcium as Ca | mg/L | 169–255 |
| Magnesium as Mg | mg/L | 29–41 |
| Sodium as Na | mg/L | 69–76 |
| Chloride (as Cl) | mg/L | 600–905 |
| Sulphate (as SO ₄) | mg/L | 65–78 |
| Nitrate | mg/L | 12–27 |
| Phosphate | mg/L | 72–86 |
| Potassium as K | mg/L | 415–690 |
| Oil and grease | mg/L | 18–32 |

2.4. Anaerobic digester: experimental set-up

The RMW and distillery sludge were loaded into the anaerobic digester. The size of the batch digester was designed such that its working volume was 3.5 L. The effective working volume of the digester was maintained as 25% of the distillery sludge and 75% of the RMW loaded

at a mesophilic temperature for the study purpose. The anaerobic digester consists of a sample loading port on the top view through which samples (distillery sludge and RMW) are added, and the digester stirred using a magnetic stirrer. Biogas production starts after 15 days from the date of adding the digester inoculum (Feng *et al.*, 2018). After achieving uniform biogas production for subsequent three days, the loading started, and the biogas production monitored.

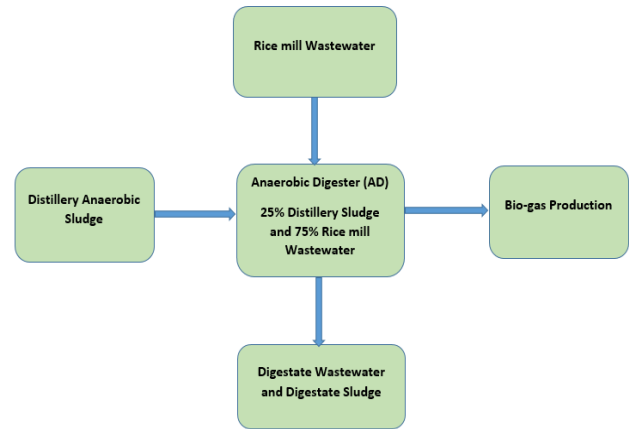


Figure 1. Block diagram of biogas production from rice mill wastewater

Constant stirred tank reactor was kept up at ambient room temperature; it has a diameter of 140mm across a height of 275 mm. Manual feeding was conducted semi continuously through the feeding hole (5 mm in diameter), which is close to the bottom, by means for a feeding port reached out to the bottom. Test samples were collected from the examining port (5 mm in width) located 130 mm from the bottom as well as from the top flexible hose extended to the bottom with a stopcock. Blending was accomplished by mixing the medium with a magnetic bar (0.1 cm in diameter and 5 cm in length) model 5 MLH plus supplied by Remi laboratory Instruments, Maharashtra, India.

The stable condition was maintained for 15 days of digestion. Figure 2 shows the bioreactor used for the experimental analysis, depicting the sample loading port as well as the collection of biogas and the treated effluent. From the reactor, the produced biogas was collected through a gas pipeline and stored in the gas displacement bottle. The percentage of biogas produced per day was examined using a gas measuring cylinder.

2.5. Operating conditions

After 15-day stabilization of the anaerobic digester, the biogas production started. After ensuring the stabilization, the OLR started and each OLR was skipped to the next loading after ensuring 3 days of constant biogas production. The proposed study was divided into three phases, namely phase I (effect of varying OLR and constant HRT), phase II (effect of varying HRT and constant OLR) and phase III (effect of both varying OLR and HRT), based on the increasing and decreasing feed concentration (Veluchamy and Kalamdhad, 2017). On the basis of OLR and

HRT conditions, biogas production was monitored on a daily basis; the percentage of biogas production, reduction of COD in biogas, and percentage of methane yield were determined.

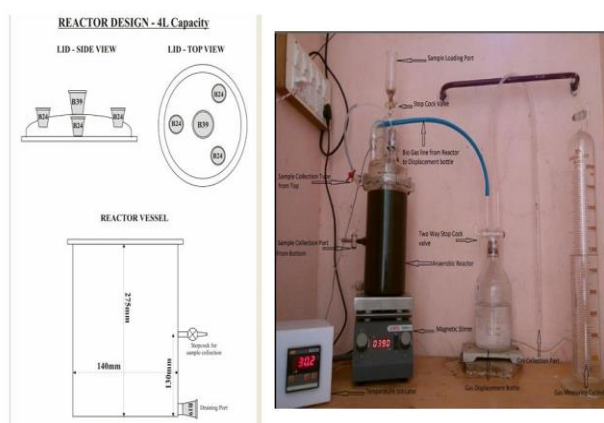


Figure 2. Experimental setup of biogas production

2.6. Chemical analyses

In the chemical analysis, a calibrated cylinder was used for measuring the gas production volume. Moreover, biogas was analyzed as per the Indian Standard (IS 13270:1992) by using the Orsat method. Then, the direct titration method was used to measure alkalinity. On the basis of these standards, BOD, COD, total volatile fatty acids, pH, total solids, total suspended solid, total kjeldahl nitrogen, volatile suspended solids, and oil and grease were analyzed using the APHA 2012 standard methods. All the samples were analyzed in triplicate and average values are reported.

Table 2. (a) Phase I: Varying OLR (0.25 to 3.91) and HRT (1 day) constant. (b) Phase II: Varying HRT (2 to 32 days) and OLR (2.5) constant. (c) Phase III: Varying OLR (0.25 to 3.75) and HRT (2 to 32 days)

| (a) | | | | | | | | |
|----------------------------------|--------|-------|-------|-------|-------|-------|--------|-------|
| OLR (kg COD/m ³ /day) | 0.52 | 1.04 | 1.56 | 2.08 | 2.61 | 3.13 | 3.65 | 3.91 |
| Biogas production(mL/day) | 337 | 680 | 931 | 1200 | 1413 | 1650 | 1816 | 1900 |
| Biogas yield(L/gm COD) | 0.209 | 0.220 | 0.208 | 0.211 | 0.210 | 0.205 | 0.200 | 0.20 |
| Methane production(mL/day) | 175 | 340 | 440 | 510 | 560 | 610 | 620 | 722 |
| Methane yield(L/gm COD) | 0.108 | 0.110 | 0.098 | 0.090 | 0.083 | 0.076 | 0.068 | 0.076 |
| COD reduction efficiency (%) | 88 | 85 | 82 | 78 | 74 | 73 | 71 | 69 |
| (b) | | | | | | | | |
| OLR (kg COD/m ³ /day) | 2.5 | | | | | | | |
| HRT (days) | 4 | 8 | 12 | 16 | 20 | 24 | 28 | 32 |
| COD (%) reduction | 47 | 56 | 66 | 74 | 79 | 85 | 88 | 90 |
| Biogas production(mL) | 1018 | 1188 | 1486 | 1667 | 1745 | 1775 | 1860 | 1945 |
| Biogas yield(L/gm COD) | 0.249 | 0.244 | 0.259 | 0.26 | 0.254 | 0.241 | 0.243 | 0.249 |
| Methane production(mL) | 509 | 594 | 757 | 850 | 942 | 958 | 967 | 991 |
| Methane yield(L/gm COD) | 0.124 | 0.122 | 0.132 | 0.132 | 0.137 | 0.130 | 0.126 | 0.127 |
| (c) | | | | | | | | |
| OLR (kg COD/m ³ /day) | 0.5 | 1 | 1.5 | 2 | 2.5 | 3 | 3.5 | |
| HRT (days) | 4 | 8 | 12 | 16 | 20 | 24 | 30 | |
| COD (%) reduction | 89 | 86 | 84 | 81 | 79 | 78 | 77 | |
| Biogas production(mL) | 352 | 690 | 950 | 1250 | 1490 | 1710 | 1925 | |
| Biogas yield(L/gm COD) | 0.23 | 0.25 | 0.23 | 0.25 | 0.255 | 0.23 | 0.21 | |
| Methane production(mL) | 204.16 | 400.2 | 551 | 725 | 864.2 | 991.8 | 1116.5 | |
| Methane yield(L/gm COD) | 0.115 | 0.115 | 0.1 | 0.092 | 0.085 | 0.078 | 0.072 | |

3. Result and discussion

3.1. Effect of varying OLR and HRT parameters

OLR: The OLR indicates the number of soluble and particulate organic matter fed into a digester per day under semi-continuous feeding. With the increasing OLR, the biogas yield increases to an extent; however, the equilibrium and productivity of the digestion process can also be considerably disturbed.

HRT: The retention time is the time required to complete the degradation of organic matter. The HRT is associated with the microbial growth rate and depends on the process temperature, OLR, and substrate composition.

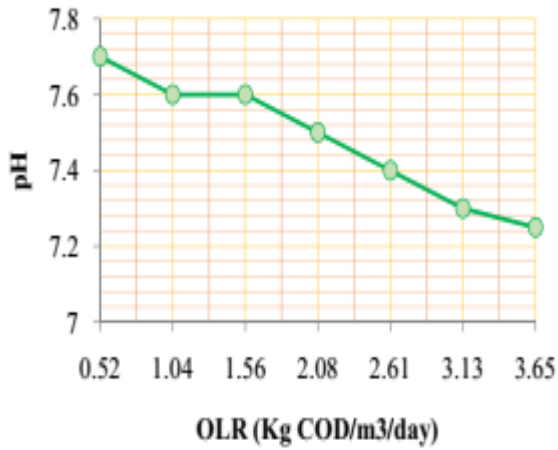
Under steady-state conditions, the mesophilic anaerobic digestion was performed using progressive OLRs and HRT; the parameters such as pH, alkalinity, VFA, biogas production, COD reduction, and methane yield were investigated by optimizing the conditions of the OLR and HRT.

3.2. Experimental results given in below Table 2

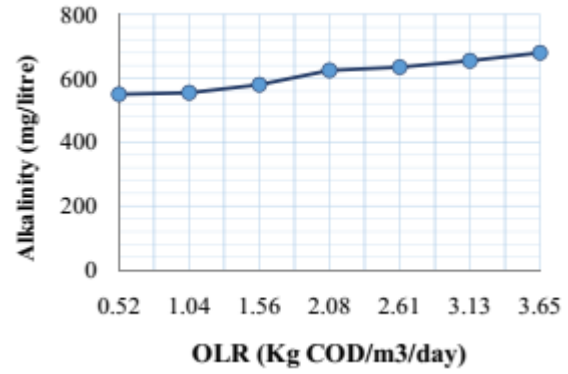
Table 2 lists the experimental results of RMW treatment with an anaerobic digester, and its yields are illustrated for three different phases. In Phase I, the OLR was varied from 0.25 to 3.91, and the results obtained are depicted in Table 2. When the OLR was increased, the methane yield decreased slowly. In phase II, the OLR was kept constant at 2.5 for the entire HRT (2–32 days), and biogas production was examined under conditions of varying HRT. In phase III, the OLR was varied from 0.25 to 3.75, and the HRT was varied from 2 to 32 days; the highest efficiency of COD reduction was 89%, which was achieved in phase III.

3.3. The behavior of pH, alkalinity, and VFA in each phase

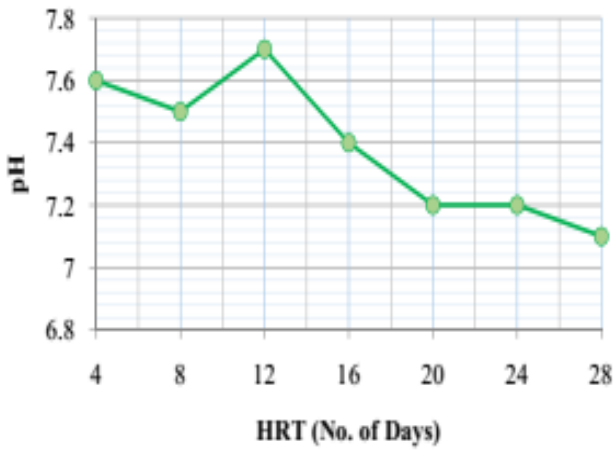
The pH value was slightly acidic in all the phases; it gradually reached an optimal value. Similarly, Figure 3 (b) and (c) explain the pH range of phase II and phase III, respectively. In phase III, the highest pH value of 7.7 was achieved under three conditions (an OLR of 0.25 and HRT of 2 days, OLR of 0.75 and HRT of 6 days, and OLR of 0.75 and HRT of 12 days).



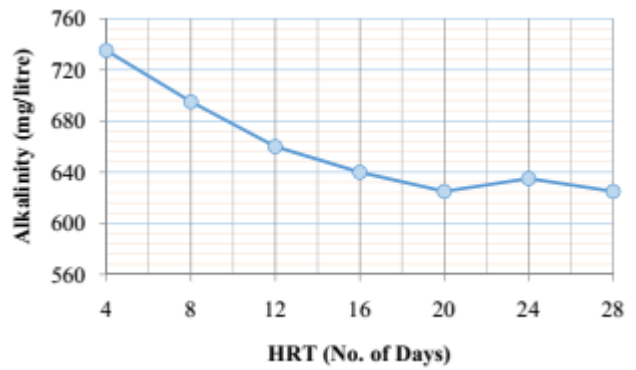
(a)



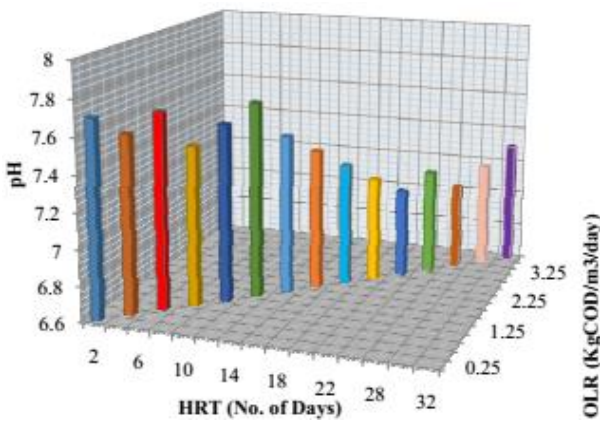
(a)



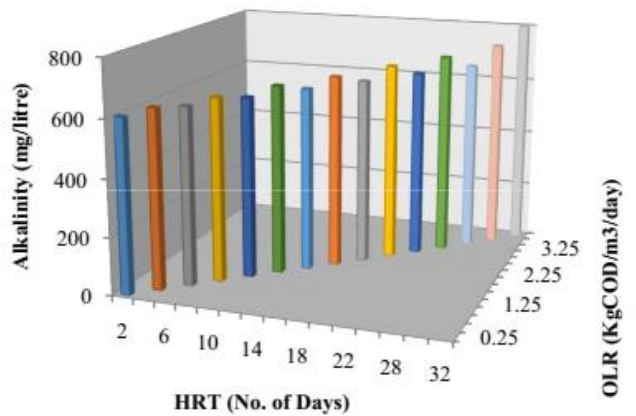
(b)



(b)



(c)



(c)

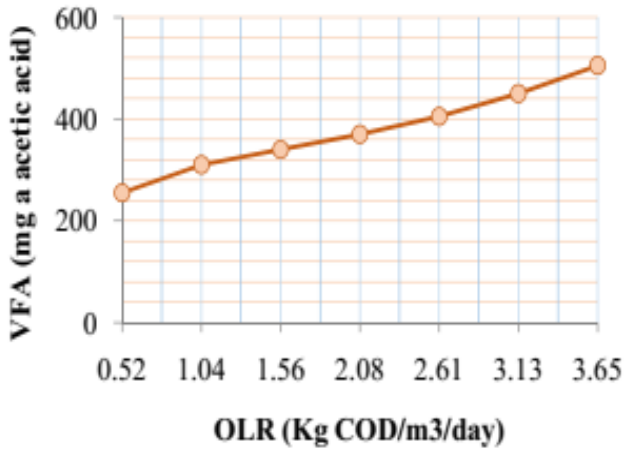
Figure 3. pH variation (a) phase I (b) phase II (c) phase III

Figure 4. Alkalinity variation (a) phase I (b) phase II (c) phase III

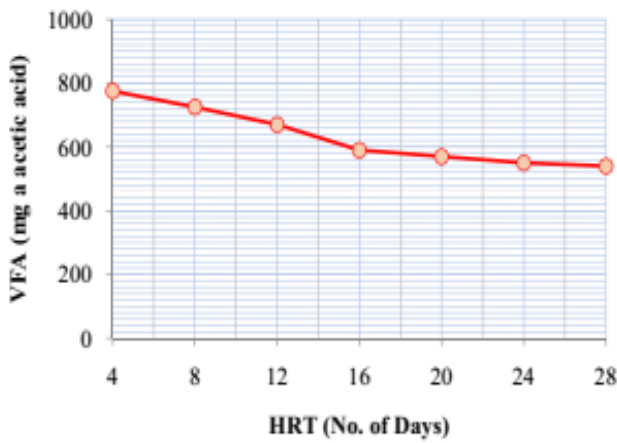
Figure 3 shows the pH variation in RMW treatment with an anaerobic digester. The pH of the produced biogas was monitored and is depicted in the graph. Figure 3 (a) shows that the pH value was 7.7 when the OLR was 0.52 in phase I, and it gradually decreased with the increasing OLR.

During the RMW treatment, the alkalinity variation was analyzed, and the results are shown in Figure 4 for the three different phases. The total alkalinity of the RMW was in the range of 500 to 750 mg/L. The alkalinity of the

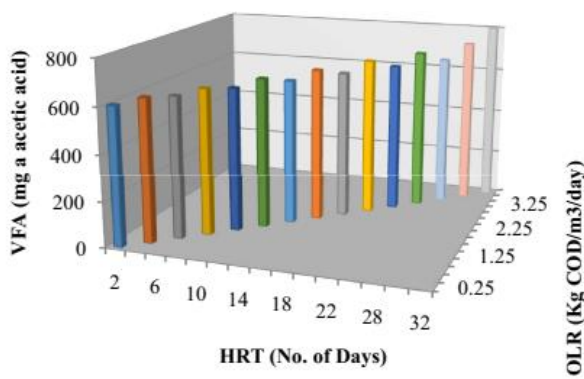
reactor in phases I, II, and III is shown in Figure (a), (b), and (c); it ranged from 550 to 680, 625 to 735, and 625 to 735 mg/L, respectively. The total alkalinity of the reactor slightly decreased with time.



(a)



(b)



(c)

Figure 5. VFA variation (a) phase I (b) phase II (c) phase III

The VFA variation was analyzed under phase I, phase II, and phase III based on the increasing and decreasing feed concentration; the results are shown in Figure 5. Acidogenic conditions at the front compartments converted fatty acids, amino acids, and monosaccharides into simple

organic acids. An increase in pH was observed in consecutive compartments because of the reduction in the VFA concentration and an increase in alkalinity. Methanogens convert acetic acid and hydrogen to methane and carbon dioxide, causing an increase in pH.

3.4. Analysis of biogas production and methane yield under three different phases

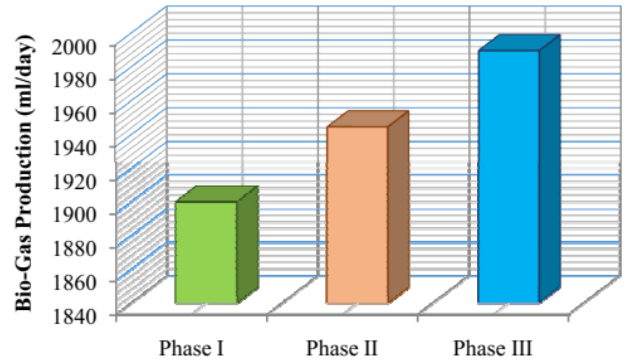


Figure 6. Biogas production

The biogas production from RMW treatment under different loading rates and time periods is shown in Figure 6. After 15-day stabilization of the anaerobic digester, initially the fluctuation was observed in biogas production for week period, and then production was uniform for three days. Finally, the produced gas was monitored, and the values are listed in Table 1. In phase 1, the OLR was varied from 0.26 to 3.91 and HRT was kept constant at 1 day; for every loading rate, its corresponding gas production was determined. The bar graph shows the highest production in phase I under the loading condition of an OLR of 3.91. In phase II, HRT was varied from 2 to 32 days, and OLR was kept constant at 2.5. The highest production of biogas was observed at 32 days of hydraulic retention, which is plotted in the graph. In phase III, HRT was varied from 2 to 32 days, and the OLR was varied from 0.25 to 3.75; the highest production of biogas was observed at an HRT of 32 days and an OLR of 3.75, which is plotted in the graph. These results indicated that the maximum production of biogas was achieved at an increased OLR and HRT.

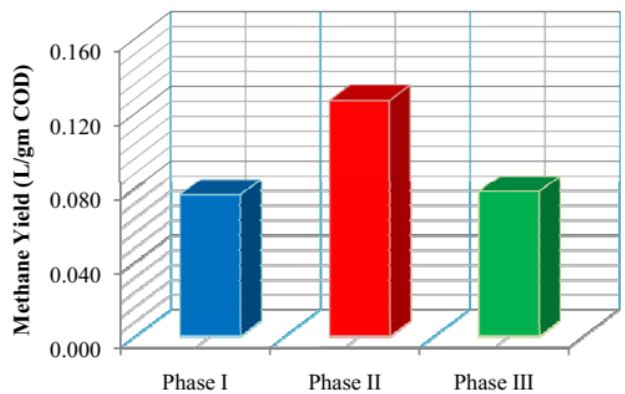
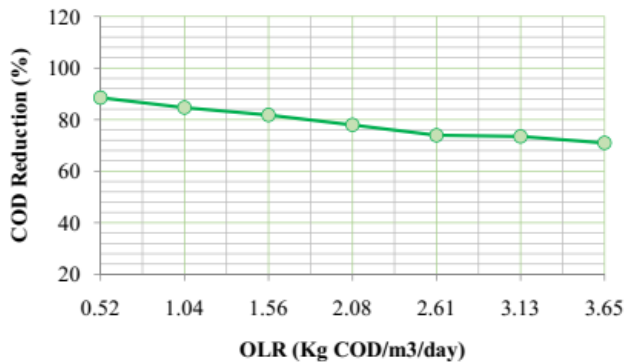
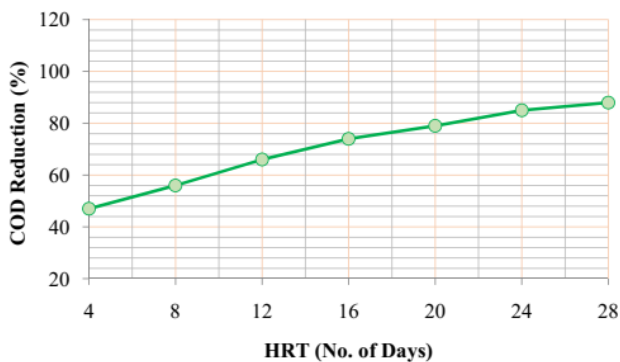


Figure 7. Methane yield

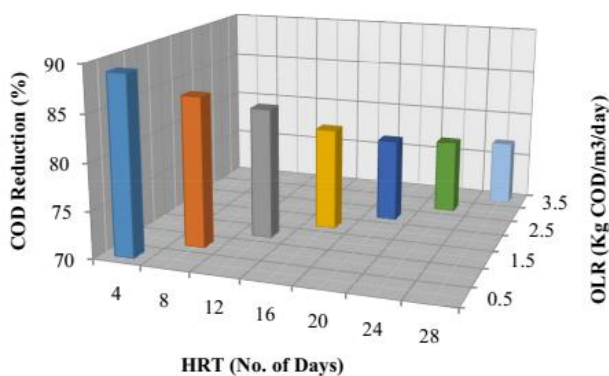
Methane production is another essential factor for evaluating the suitability of an anaerobic digester. The correlation between CH_4 yield ($\text{L CH}_4/\text{g COD day}$) and OLR and HRT is shown in Figure 7. Throughout the operation process, the CH_4 yield significantly increased from 0.123 to 0.14 $\text{L CH}_4/\text{g COD day}$. As observed in Figure 7, the CH_4 yield decreased after every change in the OLR but recovered shortly. These results indicated that the CH_4 yield is a function of the OLR condition. A low OLR resulted in low substrate contents inside the reactor, which eventually resulted in a low CH_4 production rate. On comparing the three conditions, phase II achieved the maximum methane yield.



(a)



(b)



(c)

Figure 8. COD reduction efficiency (a) phase I (b) phase II (c) phase III

3.5. Efficiency of COD reduction

The COD reduction efficiency of RMW treatment is shown in Figure 8. Different organic loadings of 0.26 to 3.91 $\text{kg COD}/\text{m}^3/\text{day}$ were studied at an HRT of 1 day (phase I). Different HRT ranging from 2 to 32 days was studied at a constant OLR of 2.5 (phase II and phase III; i.e., the effect of varying both OLR and HRT). Figure 8 (a) shows the efficiency of COD reduction in phase I, where the efficiency decreased for the maximum loading rate. Figure 8 (b) shows the efficiency of COD reduction in phase II; at an increased HRT, the reduction of COD decreased; hence, its efficiency was improved. Likewise, as shown in Figure 8 (c), in phase III, the highest efficiency was obtained at an OLR of 0.5 and an HRT of 4 days.

4. Conclusion

This study proposed AD treatment for biogas production from RMW. The results of the study indicated that anaerobic treatment is a favorable option for RMW treatment. The highest COD reduction efficiency of 91% was observed in phase III. In addition, the highest biogas production was obtained at an HRT of 32 days and an OLR of 3.75. VFA and COD reduction showed the same pattern during rice mill effluent treatment. During the study period, the reactor worked very efficiently. Regular biogas monitoring was performed, and under varying conditions of OLR and HRT, biogas production, methane yield, and percentage of COD reduction were examined. In summary, a low OLR and a long HRT provide the best strategy for achieving a constant and maximal methane yield. In the future, solid and liquid digestate derived from the anaerobic digestion of RMW can be used for algae cultivation as a substrate because of its rich plant nutrients, besides it can improve the performance of the biological degradation of industrial effluents.

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