An Experimental Study on Biogas production by Anaerobic Digestion of Rice Mill Wastewater
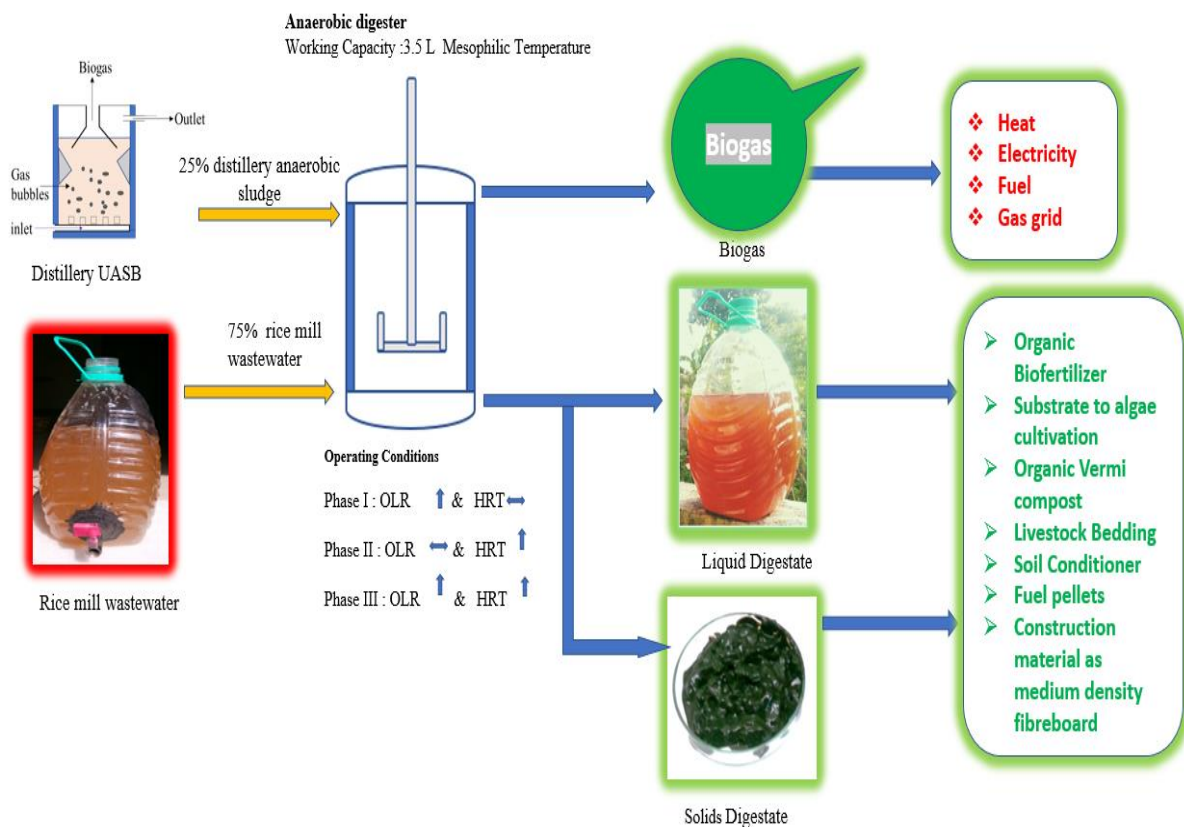
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Graphical abstract

Abstract: With the rising interest for renewable power source and ecological security, anaerobic digestion of biogas technology has attracteda considerable attention amongresearchers. This study proposed a new technique for producing biogas from ricemill wastewater (RMW) by utilizing an anaerobic digester. An anaerobic digester waspacked with RMW and distillery anaerobic sludge at a mesophilic temperature for 15 days forstabilization. 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 $COD/m^3/day$, and the HRT was varied from 1 to 32days. The total chemical oxygen demand utilized was >75%, and the CH_4 percentage of the biogas was 62%–63% for the OLRs studied. The efficient working volume of the digesterwas maintainedas 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 wereexamined.Thisenhanced the biological treatment of the effluent with the anaerobic sludge in a continuousactivity 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, Cano and Fdz-Polanco 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 animportantnutritious 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 (Noguchi et al., 2017, Kamali and Khodaparast 2015). 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, Verma and Nashine 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 remain sharmful because it contains a significant number of serious issues (Dhanasekar and Sasivarman 2016, Nain, Gupta and Bedi 2015). Therefore, there is a critical need todevelop new high-performance and cost-effective techniques that can efficiently treat rice mill wastewater (RMW) (Yuan et al., 2012, Hu et al., 2018). Anaerobic assimilation is considered a reasonable technique fortreating 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 (Weng, Ismail and Ahmad 2014, Haider et al.). 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). Inan anaerobic digester system, biogas can be producedfromdifferent 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 centerformicroalgal 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 highestmicroalgal development of 1074 mg volatile suspended solid(VSS)/L was obtainedwhen 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 inbetter microbial system modules than did the HRT, indicatingthe further subdivision of useful parts. The strongestpairwise link between firmicutes and biogas production demonstratedthat microbe'sdependent on the system can be considered as markers of AD.

In 2014 (Okeh, Onwosi and Odibo 2014) explored lab-scale biogas production from rice husks generated from various rice mills byusingcow 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 of30 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 (CH_4)-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 examinethe 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 CH_4 at the lowest OLR1, and the mean yield of biogas was 447mL/L/d at 52%CH_4 under the most favourable reactorconditions.

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 anambient temperature wasfound to be more stable. Bio electrochemical anaerobic absorption that can spare the thermal energy input at an ambient temperature is recommendedfor 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 parboilingof 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)/m^3/day were examined. Results demonstrated that the biochemical oxygen demand (BOD) and COD reductionranged from 83.0% to 92.7% and 80.2% to 89.0%, respectively, when Biopac media were used in the reactor systemand 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 theFugino spiral media-packed reactor.

In 2016, Saini (Saini, Saini and Lohchab 2016) treated RMWby 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% RMWfeed 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 substantialamount 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 towastewaterdischarge. The wastewater released from the parboiling process that has ahigh BOD and COD constitutes the major source of water pollution. Therefore, there is a need to develop new efficient and cost-effective technologies for treatingRMW.

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.

4. Material and methods

4.1. Chemicals

All reagents and chemicals were purchased from Merck Life Science Private Limited, Mumbai, India. All reagents, chemicalsused were in analytical grade and utilizedas such without any additional purification. Double distilled water was used for analysis and experiments purpose.

4. 2. Experimental investigation of RMWtreatment

In the proposed experimental analysis, biogas was produced from the RMW industry. Normally, waste water 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 RMWalong 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-Lwasfilled with 25% distillery anaerobic sludge and 75% RMW, and the produced biogas was monitored on daily basis.
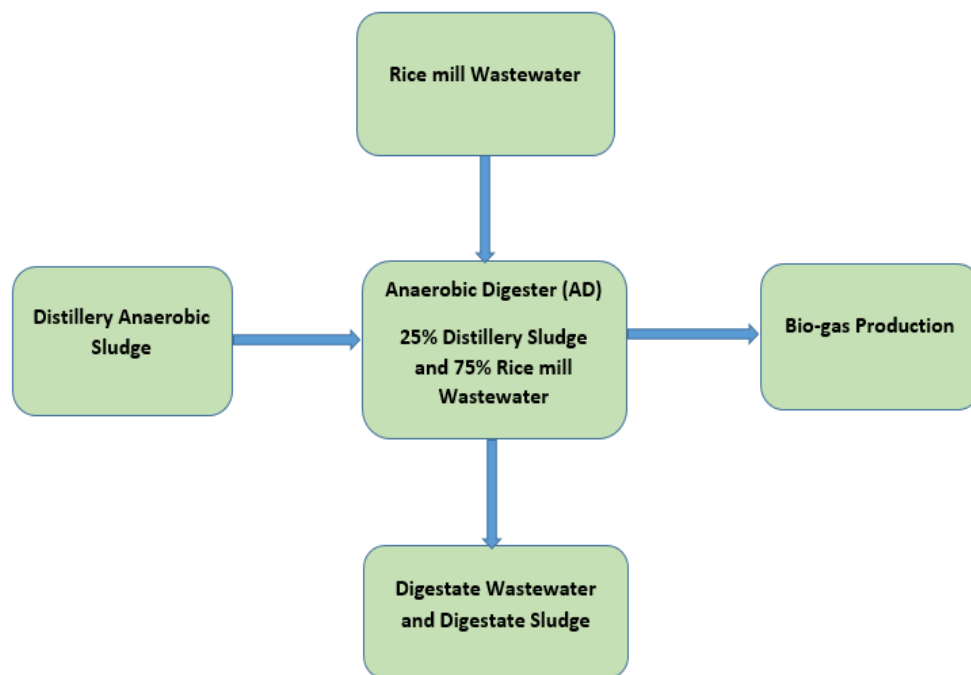

Fig 1: Block Diagram of biogasproduction from rice mill wastewater

4.3. Characterization of materials

*Inoculum***:** For the experimental analysis, the reactor was inoculated with the effluent and anaerobic sludge; that is, paddy soaking wastewatercollected from M/s Muthuraja, Modern rice mill industry, Puduvayal, Sivaganga district, Tamil Nadu, andsugar distillery anaerobic

sludge collected from EID Parry (I)Ltd., Nellikuppam, Tamil Nadu. Inthe anaerobic digester,was as follows:2.625 L (75%) of RMWand 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: 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 SO4)	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

4.4. Anaerobic digester: experimental set-up

The RMWand 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 digesterwasmaintained 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 amagnetic stirrer. Biogas production starts after 15 days from the date of adding the digester in oculum (Feng et al., 2018) . After achieving uniform biogas production forsubsequent three days, the loading started, and the biogas production monitored.

Constant stirred tank reactor were 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 thefeeding 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.
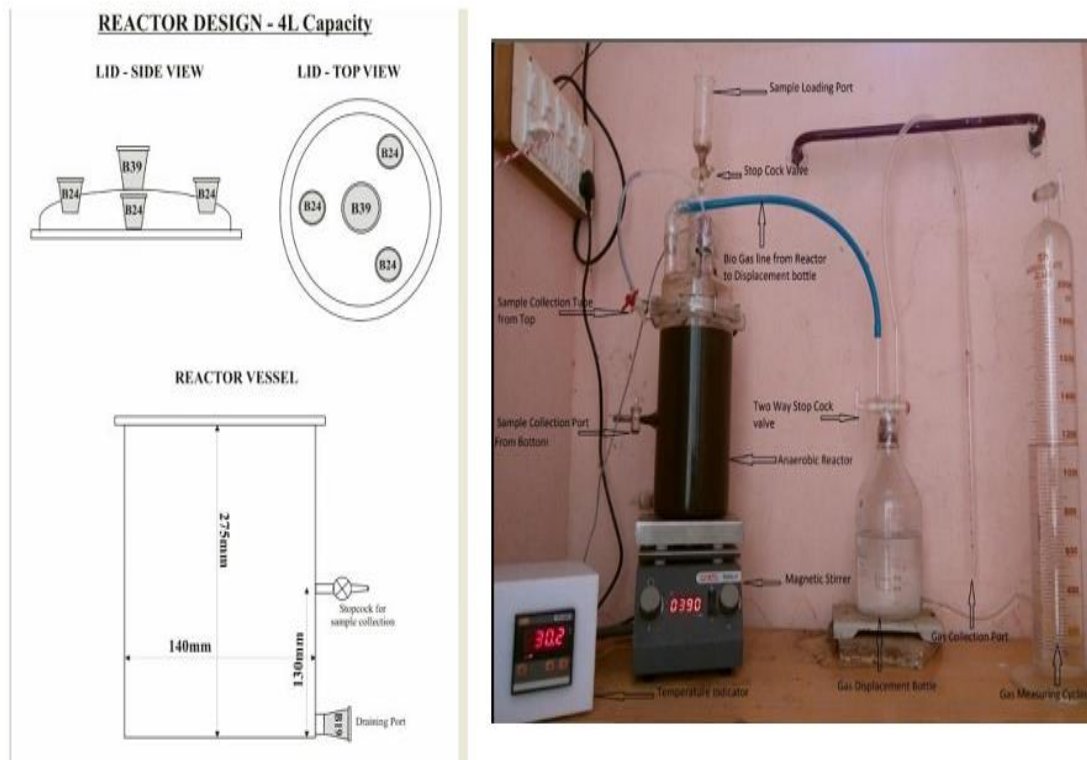
Fig 2: Experimental setup of biogas production

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 thecollection of biogas and the treated effluent. From the reactor, the produced biogaswascollected through agas pipeline and stored in the gas displacement bottle. The percentage of biogas produced per day wasexamined using a gas measuring cylinder.

4.5. 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 constantHRT), 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.

4.6. 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.

5. Result and discussion

5.1. 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 aspH, alkalinity, VFA, biogas production, COD reduction, and methane yield wereinvestigated by optimizing the conditions of the OLR and HRT.

5.2. Experimental results given in below table 2

(a)   *Phase I : Varying OLR (0.25 to 3.91) and HRT (1 day) constant*

OLR (kg COD/m³/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) *Phase II: Varying HRT (2 to 32 days) and OLR (2.5) constant*

OLR (kg COD/m³/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)   Phase III : Varying OLR (0.25 to 3.75) and HRT (2 to 32 days)*

OLR (kg COD/m³/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

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. Inphase II, the OLR was kept constantat 2.5 for the entire HRT (2–32 days), and biogas production was examined under conditions of varying HRT. In phase III, the OLR wasvaried from 0.25 to 3.75, and the HRT was varied from 2 to 32 days; the highest efficiency of COD reduction was89%, which was achieved in phase III.

### 5.3.The behavior of pH, alkalinity, and VFA in each phase
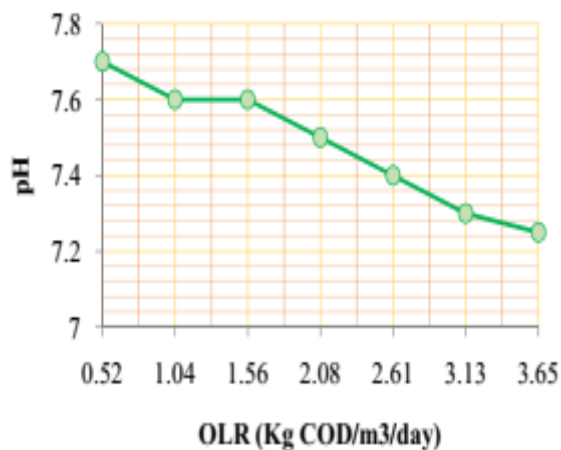
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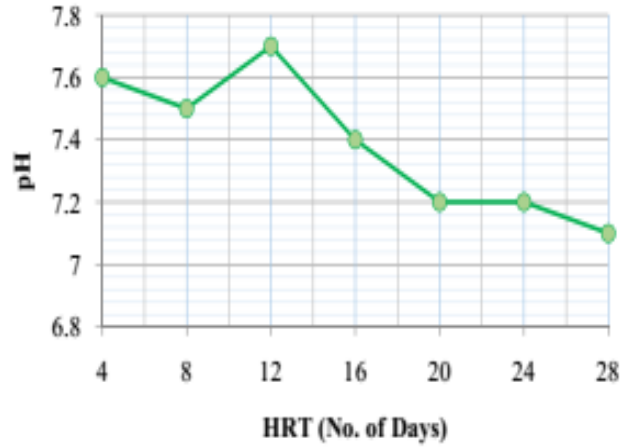
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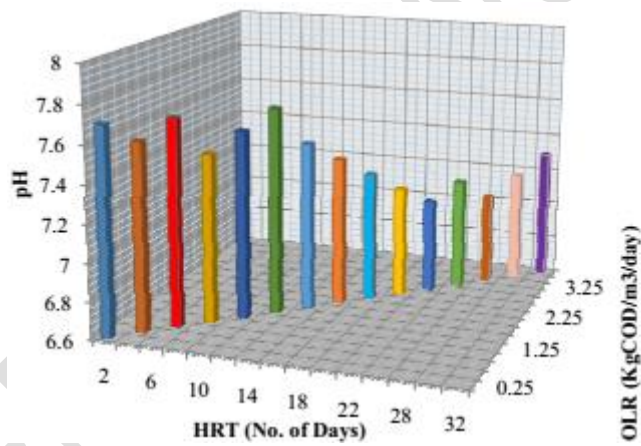
(c)

Fig 3: pH variation (a) phase I (b) phase II (c) phase III

Figure 3 shows the pH variation inRMW treatment with an anaerobic digester. The pH of the produced biogas wasmonitored 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 decreasedwiththe increasing OLR. The pH value wasslightly acidic inall 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 wasachieved 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).
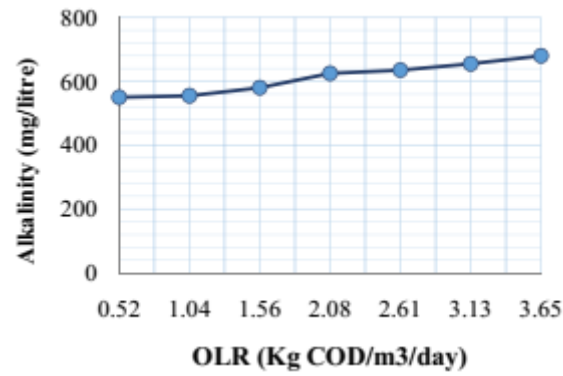

(a)
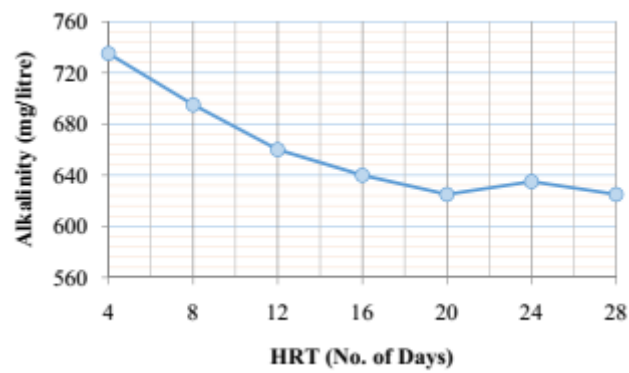
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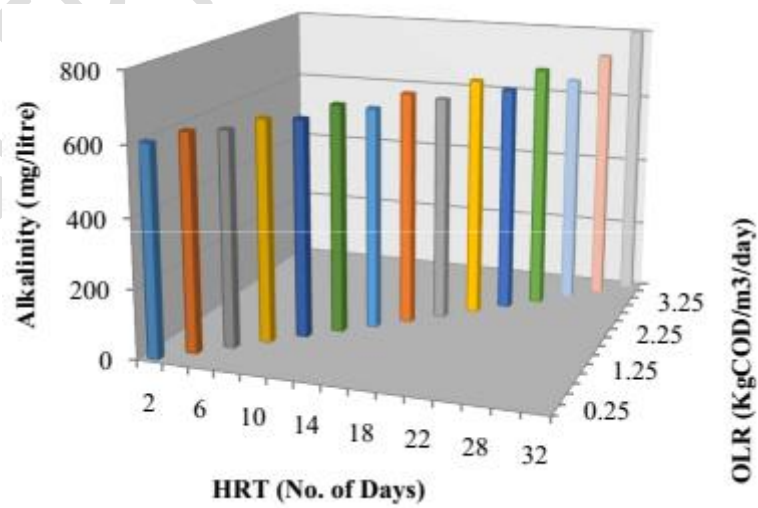
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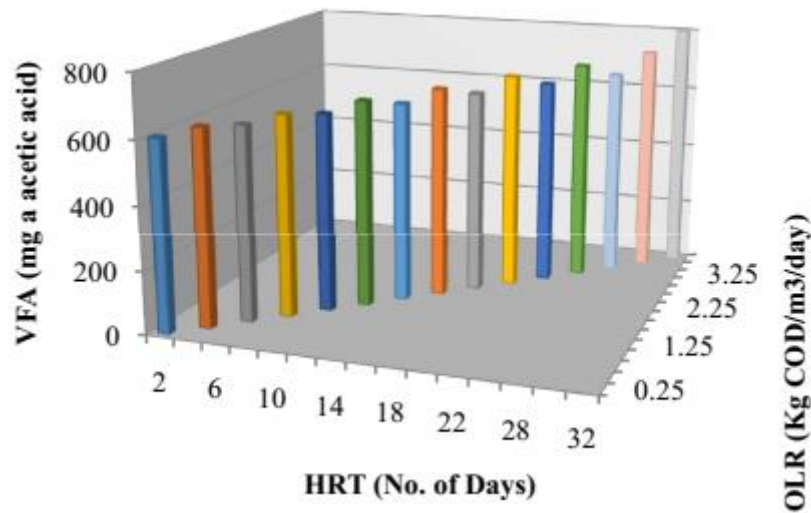**Fig 4: Alkalinity variation (a) phase I (b) phase II (c) phase III**

During the RMW treatment, the alkalinity variation wasanalyzed, 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 isshown 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.
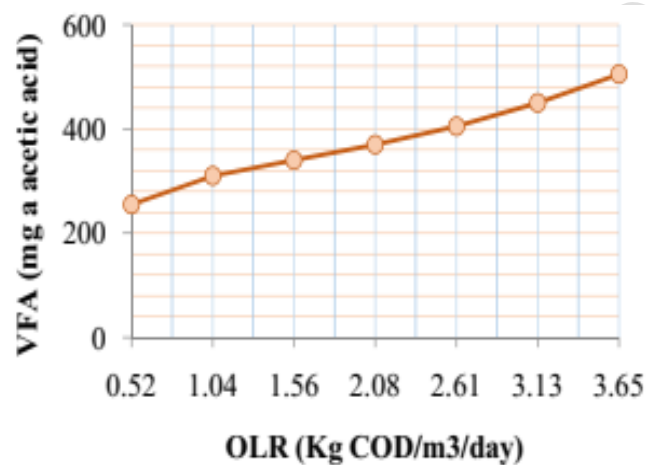
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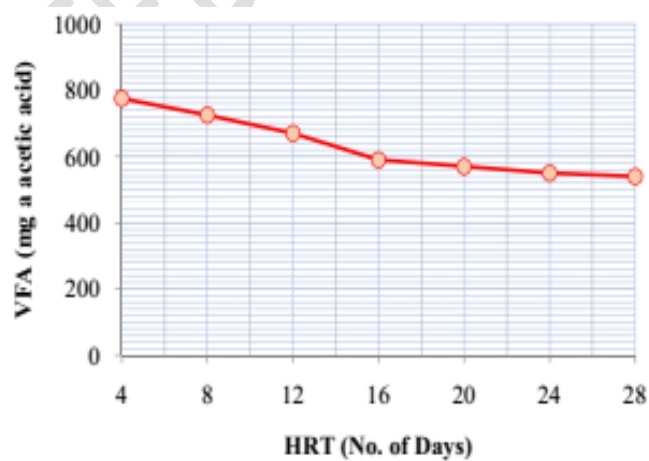
(b)

(c)

Fig 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 inFigure 5. Acidogenic conditions at the front compartments converted fatty acids, amino acids, and monosaccharidesinto simple organic acids. An increase in pH was observed in consecutive compartments because of thereductioninthe VFA concentration and an increase in alkalinity.Methanogens convert acetic acid and hydrogen to methane and carbon dioxide, causing an increase in pH.

5.4. Analysis of biogas production andmethane yield under three different phases
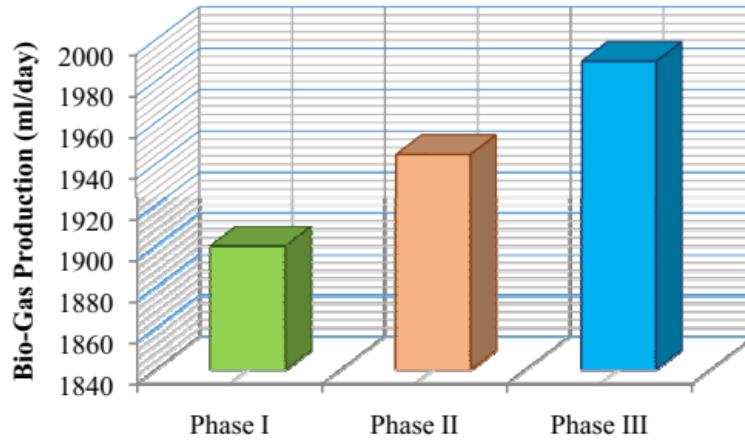
Fig 6: Biogas production

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 Table1. Inphase 1, the OLR wasvaried 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. Inphase II, HRT wasvaried from 2 to 32 days, and OLR was kept constant at 2.5.The highest production of biogas wasobserved at 32 days of hydraulic retention, which is plotted in the graph. Inphase 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 wasobserved atan 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.
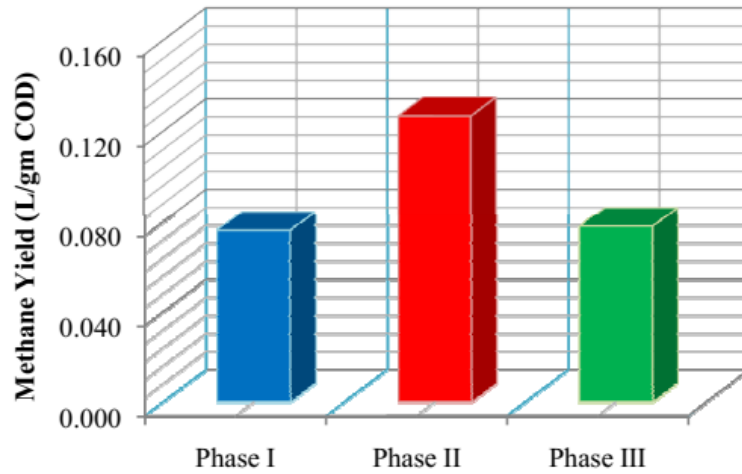
Fig 7: Methane yield

Methane production is another essential factor forevaluating the suitability of an anaerobic digester. The correlation between CH_4 yield (L CH_4/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 L CH_4/ g COD day. As observed inFigure 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.
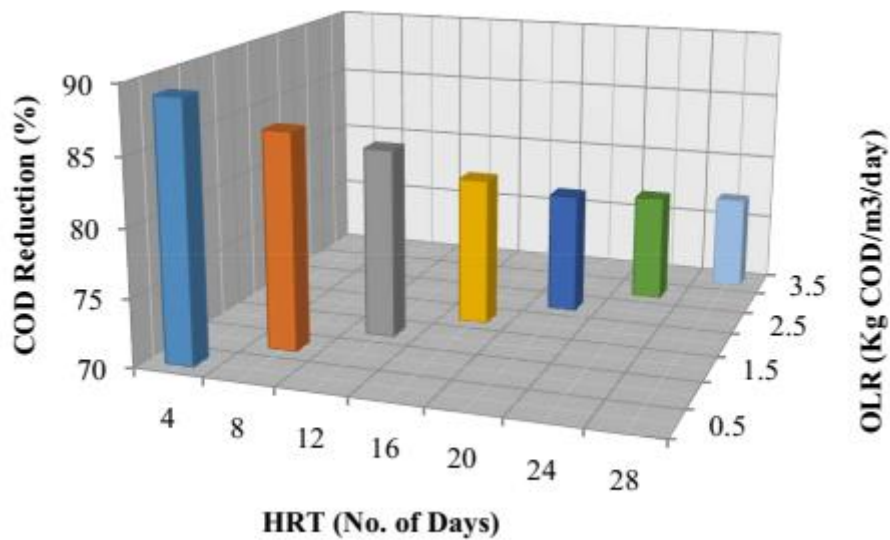
5.5.Efficiency of COD reduction
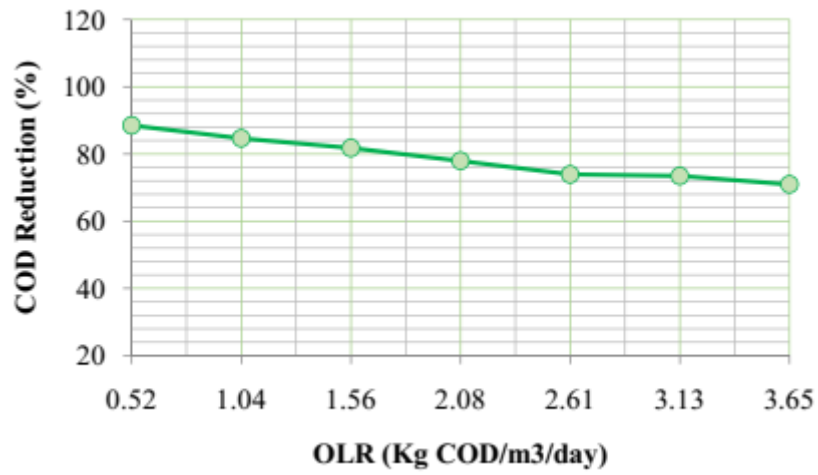
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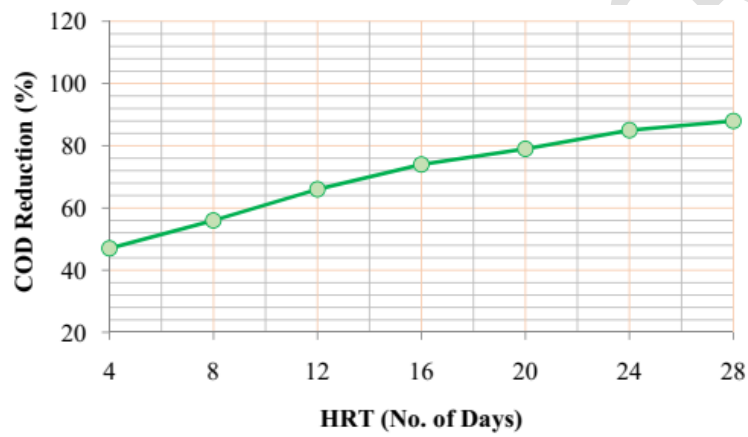
(b)

(c)

Fig 8: COD reduction efficiency (a) phase I (b) phase II (c) phase III

The COD reduction efficiency of RMW treatment is shown in Figure 8. Different organic loadings of 0.26 to 3.91kg COD/m^3/day were studied at an HRT of 1 day (phase I). Different HRT ranging from2 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 inphase I, where the efficiency decreased for the maximum loading rate. Figure 8 (b) shows the efficiency of COD reduction in phase II; atanin creased HRT, the reduction of COD decreased; hence, its efficiency wasimproved. 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.

6. 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 wereexamined.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 substratebecause of its rich plant nutrients,besides it can improve the performance of the biological degradation of industrial effluents.

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