International Journal of Pharmacy and Biological Sciences
ISSN: 2321-3272 (Print), ISSN: 2230-7605 (Online)
IJPBS | Volume 9 | Special Issue 2- ICESMT| 2019 | 01-09
International Conference on "Empowering Society with Microbial Technology"
Held at Tuljaram Chaturchand College of Arts, Science and Commerce, Baramati - 413 102,
Dist. Pune, Maharashtra, India, 7-9 February 2019
| Research Article | Biological Sciences |Open Access |MCI Approved|
|UGC Approved Journal|

The Optimization of pH and Hydraulic Retention Time (HRT) For Biomethanation of Fruits and Vegetable Waste (FVW)-Laboratory Study

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Received: 25 Jan 2019 / Accepted: 20 Feb 2019 / Published online: 01 Apr 2019
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Abstract
Fruits and vegetables (FV) are food materials most of which are readily perishable. The FV serve as foods to man, animals and serve as nutrients to microorganisms. The source of fruit and vegetable waste (FVW) includes unsold and spoiled FV, over ripened fruits, cutting and coverings of FV. The FVW produced in tones daily. Thus, FVW is admixture and mostly solid waste in nature, disposed at any available places in and around market area. In the present investigation, the mixed FVW obtained from Shree Shiva Chhatrapati Market Yard, Gultekadi Market, Pune. The waste pulverized in grinder. The biogas digesters of 5-Litre capacity were used for the studies. They were initiated using already running biogas digester effluent (BDE) and cow dung slurry (CDS). The FVW pulverized biomass was admixed with CDS and BDE; and gradually CDS and BDE replaced with FVW slurry with increments of 25, 50, 75 and 100%. The varying pH range (5.5, 6, 6.5, 7, 7.5,8 and HRT range of 20, 25, 30, 35 and 40 days) were used to optimize these parameters. It was found that at the optimum pH of slightly alkaline range (7.5) and optimum HRT of 35 days the volume of biogas generated per liter of FVW slurry (40% solids) was 16-17 liters. The bluish flame in the burning test indicated more than 55% of methane in the biogas. The results are encouraging, and biogas effluent will be further used for vermicomposting as well as generation of electricity.

Keywords
Fruits and Vegetable Waste, Gultekadi Market, Biomethanation, pH, HRT

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1. INTRODUCTION
India is leading world's agricultural produces mainly FV and stands 2nd in world's fruit production.[24] The waste generation from marketplaces in Indian cities is increasing from last few decades. The Pune alone produces daily 2000-2100 tons of solid waste out of which 7.5% is generated from marketplaces.[25] The fruits and vegetables waste (FVW) is perishable and it consist moisture. The environmental pollution is associated with market waste[1] and contains FV at various stages of degradation. The partially decomposed organic waste provides nutrients and life support to various forms of microbes susceptible to the microbial degradation[2] FVW gathered daily in the market not picked up regularly and hence the accumulated waste gets decomposed easily by

microorganism's causes environmental pollution[1,3] like filthiness, dirty conditions, bad odors, disgusting appearance in addition to which it becomes dwelling place of dogs, pigs, rodents, cats and similar nuisance creatures. The site/area is teemed with flies, insects and worms[4] and environmental degradation in terms of air, water and soil takes place.[5] Hence it is necessary to develop in-situ solid waste treatment methodology for fruit and vegetable waste at market unit level itself.[6]

The rate of generation of waste and time required for its management are inversely proportion. The hip of waste on dumping site indicates minimum or no treatment. In the vegetable and fruit market of Indian conditions, generally we observe filthy and unhygienic conditions due to fruit and vegetable wastes.[7] The ability of FVW to produce energy is well known.[8-9] The rate of biogas production from substrate is dependent on various factors like type of digester, hydraulic retention time, composition of substrate, pH, temperature.[10] In this regards present study has objective to optimize hydraulic retention time (HRT) and pH of FVW influent for biomethanation process at laboratory scale floating dome biogas digester.

2. MATERIALS AND METHODS:

2.1 Collection, processing and preservation of FVW from Gultekadi Market

The collection of FVW carried out on regular basis from Gultekadi Market Pune. The empty plastic bags used for collection. The FVW carried to laboratory and segregated from other wastes. FVW was chopped in laboratory and further it was grinded in college canteen for size reduction. The FVW was stored in deep freeze at 4°C till its use.

Fig No I: Collection, processing and preservation of FVW from Gultekadi Market

I. a) FVW collection

I.b) FVW in market

I. c) Segregation of FVW

I. d) Cutting of FVW

I. e) Grinding

2.2 Physicochemical analysis of fruit and vegetable waste and digester effluent:

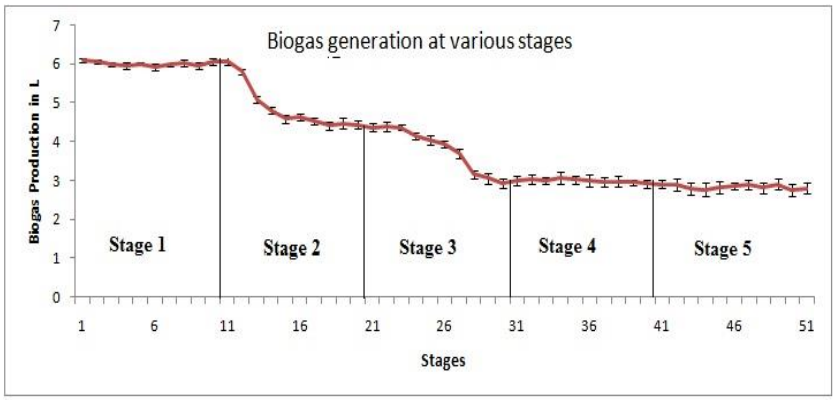
The waste analyzed after composite sampling, mixing, macerating and admixing to parameters like pH, COD, BOD, TOC, N, P, C/N, Total Solids and Total Volatile Solids. Standards procedures were used for analysis of waste.[11]

2.3 Design of Biomethanation plants (5L Capacity)

The biogas plants of 5L capacity were constructed from local fabricator. The design of laboratory scale plant was based on floating dome type anaerobic digester.[12-13] The inlet and outlet provisions were made in design. Feed was provided from inlet and slurry was collected through outlet. The provision was made to collect, and measure biogas generated in dome through small opening at center of dome, connected with pipe. The water displacement method was used for measuring gas production.[14] The methane fraction was monitored by gas chromatography.

Fig No II: Biogas Digester of 5L capacity

Fig No III: Biogas generation at different charge pattern of FVW admixed with CDS and BDE in the biogas digesters



2.4 Methodology for initiation of biomethanation

The 5L capacity locally fabricated six floating dome digesters were used for this experiment. The biomethanation was achieved by using previously running Biogas Digester Effluent (BDE) and Cow Dung Slurry (CDS) in 50:50 proportion, with 40% solids each as inoculums. The start-up was with 100% CDS and BDE as feed. The daily loading was 167 mL (CDS 83.5 mL+ BDE 83.5mL) for 30 days.

2.5 Methodology for Biogas generation at different charge pattern of FVW admixed with CDS and BDE in the biogas digesters

This experiment has five stages and each stage was of 10 days in continuation with previous stage. The Stage-I was from day 1 to 10, Stage-II was from day 11 to 20, Stage-III was from day 21 to 30, Stage-IV was from day 31 to 40 and Stage-V was from day 41 to 50. The gradual replacement of CDS and BDE with FVW was carried out in all six digesters. The proportion of CDS and BDE: FVW was 100: 0, 75: 25, 50: 50, 25: 75, and 0: 100 in Stage I to Stage V respectively (**Table No I**). The substrate was with 40% solids, pH and temperature was maintained around 7.0 and 35 to 40°C respectively. The biogas was monitored regularly during experiment.

Table No: I: Initiation of biomethanation Stages, Days and Influent composition and proportion.

STAGES	STAGE I	STAGE II	STAGE III	STAGE IV	STAGE V
Days	1 to 10	11 to 20	21 to 30	31 to 40	41 to 50
Influent composition (mL)	CDS + BDE 100%	(CDS + BDE) 75% + 25% FVW	(CDS + BDE) 50% + 50% FVW	(CDS + BDE) 25% + 75% FVW	FVW 100%
CDS	83.5	62.6	41.75	20.87	0
BDE	83.5	62.6	41.75	20.87	0
FVW	0	41.75	83.5	125.25	167

2.6 Methodology for Optimization of HRT

The 5 L capacity six digesters of previous experiment running on 100% FVW were used for optimization of HRT. The experiment was carried out in triplicate. The digesters were labeled as A, B, C, D, E, and F. The HRT was optimized considering range of 15, 20, 25, 30, 35 and 40 days. The substrate was with 40% solids, pH and temperature was maintained around 7.0 and 35 to 40°C respectively. The experiment was continued for 50 days. The feed provided to each digester was 333, 250, 200, 167, and 143, 125 mL/day for 15, 20, 25, 30, 35 and 40 days of HRT respectively **(Table No II)**. The biogas produced and methane fraction was measured regularly for all digesters.

Table No: II: Optimization of HRT: Digesters, HRT and influent

Digester	A	B	C	D	E	F
HRT (days)	15	20	25	30	35	40
Influent (mL)	333	250	200	167	143	125

2.7 Methodology for Optimization of pH for FVW influent

The six digesters viz. A, B, C, D, E, and F of 5 L capacity with optimized HRT of 35 days but the experiment was continued for 50 days for optimization of pH in three sets. The influent pH optimization carried out in the range of 5.5, 6, 6.5, 7, 7.5 and 8 **(Table No III)** and temperature was maintained around 35-40°C. The feed provided to each digester was 143 mL/day. The biogas produced and methane fraction was measured regularly for all digesters.

Table No: III: Optimization of pH: Digester, HRT and Influent

Digester	A	B	C	D	E	F
HRT (days)	35	35	35	35	35	35
Influent (mL/Day)	143	143	143	143	143	143

2.8. The efficiency of biogas digester for percentile reduction in physicochemical characteristics FVW influent and effluent.

The average minimum and maximum values for influent and effluent were recorded during the experiment. The percentile reduction in physicochemical characteristics viz. pH, TS, TVS, COD, BOD, N, P, and TOC were investigated.

3. RESULTS:

The many researchers have studied anaerobic digestion of FVW.[7, 15-16] The present work has focus on optimization of HRT and pH for FVW.

3.1 Initiation of biomethanation using CDS and BDE (50:50) as inoculum.

The experiment with six digesters was started for initiation of biomethanation using CDS and BDE (50:50) as inoculums with 40% solids in it at 5-L digester. Biogas production was observed between 2.5±0.01 to 6.1 ± 0.02 per liter of influent. The biogas production increased from 1st day to 10th day from 2.5 to 6.0 L. It was constant for 11th and 12th day, form 13th day to 30th day it was between 5.8 to 6.1 L/day. The total biogas production for 30 days experiment was 165.8 L with average production of 5.5L/day. The burn test was positive for all days indicating methane in it (Table No IV).

Table IV: Initiation of biomethanation using CDS and BDE (50:50) as inoculum.

Day	Average Biogas production of six digester (L)	*Burn Test
1	2.5 ± 0.01	YES
2	3.0 ± 0.01	YES
3	3.8 ± 0.01	YES
4	4.4 ± 0.01	YES
5	4.7± 0.01	YES
6	5.1± 0.02	YES
7	5.4± 0.01	YES
8	5.7± 0.01	YES
9	5.9± 0.02	YES
10	6.0± 0.01	YES
11	6.0± 0.01	YES
12	6.0± 0.01	YES
13	5.9± 0.01	YES
14	5.8± 0.01	YES
15	5.8± 0.01	YES
16	5.9± 0.02	YES
17	5.9± 0.01	YES
18	5.9± 0.01	YES
19	6.0± 0.01	YES
20	5.9± 0.01	YES
21	6.0± 0.02	YES
22	6.1± 0.01	YES
23	5.9± 0.01	YES
24	6.0± 0.01	YES
25	6.1± 0.02	YES
26	6.1± 0.01	YES
27	6.0± 0.02	YES
28	5.9± 0.01	YES
29	6.0± 0.01	YES
30	6.0± 0.02	YES

*Burn Test: It was positive and yellowish blue flame reported

Table No. V: Biogas generation at different charge pattern of FVW admixed with CDS and BDE in the biogas digesters

STAGES	STAGE I	STAGE II	STAGE III	STAGE IV	STAGE V
1	6.1 ± 0.01	5.8 ± 0.03	4.4 ± 0.04	3.0 ± 0.03	2.9 ± 0.02
2	6.0 ± 0.02	5.1 ±0.02	4.4 ±0.04	3.0 ± 0.03	2.8 ± 0.03
3	6.0 ± 0.02	4.8 ± 0.04	4.1 ± 0.02	3.1 ± 0.02	2.8 ± 0.01
4	6.0 ± 0.01	4.6 ± 0.01	4.0 ± 0.01	3.0 ± 0.05	2.8 ± 0.04
5	5.9 ± 0.02	4.6 ± 0.03	3.9 ± 0.05	3.0 ± 0.02	2.9 ± 0.01
6	6.0 ± 0.01	4.5 ± 0.04	3.7± 0.02	3.0 ± 0.01	2.9 ± 0.04
7	6.0 ± 0.03	4.4 ± 0.01	3.2 ± 0.01	3.0 ± 0.01	2.8 ± 0.02
8	6.0 ±0.02	4.5 ± 0.01	3.1 ± 0.01	3.0 ± 0.04	2.9 ± 0.02
9	6.1 ± 0.02	4.4 ± 0.02	2.9 ± 0.02	2.9 ± 0.03	2.8 ± 0.01
10	6.1 ± 0.03	4.4 ± 0.04	3.0 ± 0.02	2.9 ± 0.03	2.8 ± 0.01
Total	60.07 ± 0.02	47.21 ± 0.03	36.72 ± 0.02	29.84 ± 0.03	28.36 ± 0.02
Average	6.01 ± 0.02	4.72 ± 0.03	3.67 ± 0.02	2.98 ± 0.03	2.84 ± 0.02

Table No VI: Optimization of Hydraulic Retention Time (HRT)

Time	A (15/333)		B (20/250)		C (25/200)		D (30/167)		E (35/143)		F (40/125)	
	BG	CH4	BG	CH4	BG	CH4	BG	CH4	BG	CH4	BG	CH4
Week1	6.52 ± 0.04	1.63 ± 0.03	4.88 ± 0.01	1.69 ± 0.01	3.86 ± 0.01	1.7 ± 0.03	3.07 ± 0.05	1.60 ± 0.03	2.63 ± 0.07	1.93 ± 0.03	2.23 ± 0.04	1.64 ± 0.01
Week2	6.46 ± 0.01	1.65 ± 0.01	4.83 ± 0.03	1.72 ± 0.03	3.8 ± 0.01	1.75 ± 0.07	3.11 ± 0.02	1.71 ± 0.03	2.65 ± 0.03	1.96 ± 0.02	2.23 ± 0.01	1.64 ± 0.04
Week3	6.56 ± 0.03	1.69 ± 0.04	4.75 ± 0.05	1.72 ± 0.02	3.82 ± 0.05	1.84 ± 0.07	3.09 ± 0.02	1.85 ± 0.02	2.63 ± 0.05	1.94 ± 0.07	2.23 ± 0.01	1.65 ± 0.01
Week4	6.49 ± 0.03	1.69 ± 0.04	4.8 5± 0.07	1.88 ± 0.04	3.66 ± 0.01	1.84 ± 0.01	3.12 ± 0.04	1.97 ± 0.02	2.67 ± 0.01	1.98 ± 0.04	2.24 ± 0.02	1.66 ± 0.03
Week5	6.59 ± 0.03	1.75 ± 0.06	4.75 ± 0.04	1.9 ± 0.08	3.68 ± 0.01	1.91 ± 0.03	3.09 ± 0.03	2.04 ± 0.02	2.67± 0.09	1.98 ± 0.01	2.26 ± 0.02	1.68 ± 0.02
Week6	6.53 ± 0.04	1.74 ± 0.07	4.78 ± 0.02	2.01± 0.03	3.7 ± 0.02	2.04 ± 0.02	3.07 ± 0.01	2.12 ± 0.02	2.65 ± 0.02	1.95 ± 0.03	2.26 ± 0.01	1.68 ± 0.01
Total	39.16 ±0.03	10.15 ±0.04	28.83 ±0.04	10.93 ±0.03	22.52 ±0.02	11.08 ±0.06	18.55 ±0.03	11.29 ±0.02	15.92 ±0.05	11.74 ±0.03	13.44 ±0.02	9.96± 0.02
Average	6.53 ± 0.03	1.69 ±0.04	4.80± 0.04	1.82± 0.03	3.75± 0.02	1.85± 0.06	3.09± 0.03	1.88± 0.02	2.65± 0.05	1.96± 0.03	2.24± 0.02	1.66± 0.02

BG: Biogas, CH4: Methane

Table No VII: Optimization of pH for FVW Influent

Sr. No	Condition	Value
1	HRT	35 Days
2	pH	7.5

Table No. VIII: Optimized conditions of FVW Influent for biomethanation

Digester (influent pH)												
	A (5.5)		B (6)		C (6.5)		D (7)		E (7.5)		F (8)	
Time	BG	CH4	BG	CH4	BG	CH4	BG	CH4	BG	CH4	BG	CH4
Week1	0.80 ± 0.01	0.27 ± 0.02	1.08 ± 0.01	0.53 ± 0.01	1.80 ± 0.01	0.94 ± 0.02	2.23 ± 0.02	1.39 ± 0.01	2.70 ± 0.08	1.98 ± 0.01	2.25 ± 0.02	1.46 ± 0.01
Week2	0.76 ± 0.03	0.26 ± 0.03	1.07 ± 0.01	0.53 ±0.01	1.79 ± 0.04	0.97 ± 0.03	2.20 ± 0.01	1.43 ± 0.02	2.67 ± 0.01	1.97 ± 0.02	2.37 ± 0.01	1.53 ± 0.01
Week3	0.76 ± 0.03	0.26 ± 0.02	1.12 ± 0.03	0.55 ± 0.02	1.89 ± 0.03	1.05 ± 0.03	2.25 ± 0.01	1.49 ± 0.02	2.69 ± 0.01	1.99 ± 0.02	2.26 ± 0.02	1.46 ± 0.01
Week4	0.61 ± 0.01	0.22 ± 0.02	1.04 ± 0.01	0.52 ± 0.03	1.83 ± 0.02	1.04 ± 0.01	2.32 ± 0.05	1.57 ± 0.01	2.72 ± 0.03	2.01 ± 0.02	2.30 ± 0.01	1.50 ± 0.03
Week5	0.57 ± 0.01	0.20 ± 0.03	1.07 ± 0.06	0.55 ± 0.01	1.84 ± 0.01	1.08 ± 0.01	2.26 ± 0.02	1.57 ± 0.02	2.70 ± 0.01	2.00 ± 0.03	2.23 ± 0.02	1.46 ± 0.02
Week6	0.56 ± 0.00	0.20 ± 0.01	1.09 ± 0.01	0.56 ± 0.01	1.89 ± 0.01	1.17 ± 0.01	2.22 ± 0.01	1.56 ± 0.02	2.69 ± 0.07	1.99 ± 0.02	2.30 ± 0.04	1.51 ± 0.02
Total	4.06 ± 0.01	1.40 ±0.02	6.48 ±0.02	3.24 ± 0.02	11.04 ± 0.02	6.24 ± 0.02	13.47 ±0.03	9.02 ± 0.03	16.17 ± 0.03	11.95 ± 0.03	13.71 ± 0.03	8.91 ± 0.02
Average	0.68 ±0.01	0.23 ±0.02	1.08 ± 0.02	0.54 ± 0.02	1.84 ±0.02	1.04 ±0.02	2.25 ±0.03	1.5 ± 0.03	2.7 ± 0.03	1.99 ± 0.03	2.29 ± 0.03	1.48 ± 0.02

BG: Biogas, CH4: Methane

Table No IX: Physicochemical characteristics FVW influent and effluent

Physicochemical characteristics of FVW influent and effluent					
Sr. No	Parameter	influent Value		Effluent Value	
		Minimum	Maximum	Minimum	Maximum
1	pH	3.4	6.8	7.3	7.5
2	COD (ppm)	50400	158400	16500	42000
3	BOD (ppm)	30850	81700	8500	26000
4	N (ppm)	700	2800	150	370
5	P (ppm)	38	400	6.0	18
6	TOC (ppm)	55480	57400	9000	14000

COD: Chemical Oxygen Demand, BOD: Biochemical Oxygen Demand, N: Nitrogen: Phosphorus, TOC: Total Organic Carbon, ppm Parts per million

Table No X: Percentile reduction of physicochemical parameters

Sr.No	Parameter	% Min Reduction	% Max Reduction	Average % reduction
1	pH	NA	NA	NA
2	COD (ppm)	67.26	73.48	70.37
3	BOD (ppm)	72.45	68.18	70.31
4	N (ppm)	78.57	86.79	82.68
5	P (ppm)	84.21	95.50	89.86
6	TOC (ppm)	83.78	75.61	79.69

COD: Chemical Oxygen Demand, BOD: Biochemical Oxygen Demand, N: Nitrogen: Phosphorus, TOC: Total Organic Carbon, ppm : Parts Per million

3.2 Biogas generation at different charge pattern of FVW admixed with CDS and BDE

The total biogas production increased from Stage-I to stage-V as 60.07±0.02 and 47.21± 0.03, 36.72± 0.02, 29.84±0.03 and 28.36± 0.02 L respectively. The average biogas production for stage I to stage-V was 6.01±0.02, 4.72±0.03, 3.67±0.03, 2.98±0.03 and 2.84±0.02 L. The Stage-I showed average maximum biogas production of 6.01 ±0.02 L while Stage-V showed average minimum biogas production of 2.8±0.02 L (Table No V, Fig No: III).

3.3 Optimization of Hydraulic Retention Time (HRT)

The total biogas production has maximum value for digester A and minimum for digester F as 39.16±0.03 and 13.44±0.02 L respectively. The average maximum biogas production for Digester A was 6.53±0.03 L and that of minimum for Digester F was 2.24±0.02 L. The average maximum and average minimum methane production for Digester E and Digester F was 1.96±0.03 and 1.66±0.02 L respectively. The Digester E represents optimum HRT with highest total methane production of 11.74±0.03 L (Table No VI).

3.4 Optimization of pH for FVW influent

The total biogas production has increased trend as 4.06±0.01, 6.48±0.02, 11.04±0.02, 13.47±0.03 and 16.17±0.03 L in digester A, B, C,D, and to E respectively. The total biogas has slightly decreased in digester F as 13.71±0.03 L. The average maximum and minimum biogas production are in digester E and digester A as 2.70±0.03 and 0.68±0.01 L respectively. The digester E has shown the total maximum methane production as 11.95±0.03 and digester A has shown the total minimum methane production as 1.40 ±0.02 L. The average maximum and minimum methane production are in digester E and digester A as 2.70±0.02 and 0.23±0.02 L respectively (Table No VII). The Digester E represents optimum pH value as 7.5 with highest total methane production of 11.95±0.03 L.

3.5 OPTIMIZED CONDITIONS FOR FVW INFLUENT FOR BIOMETHANATION

The optimum HRT is 35 days and Optimum pH is 7.5 (Table No VIII)

3.6 Physicochemical characteristics of FVW influent and effluent

The influent pH showed range between 3.4 to 6.8. The influent minimum and maximum values for COD are 50400.0 and 158400.0 ppm, BOD: 30850.0 and 81700.0 ppm, nitrogen:700.0 and 2800.0 ppm, Phosphorus: 38.0 and 400.0 ppm, and TOC:55480.0 and 57400.0 ppm.

The effluent pH was between 7.3 and 7.5 respectively. The effluent minimum and maximum values for COD are 16500.0 and 42000.0ppm, BOD ppm: 8500.0 and 26000.0, Nitrogen ppm: 150.0 and 370.0, Phosphorus ppm: 6.0 and 18.0, and TOC: 9000.0 and 14000.0 ppm (Table No IX).

3.7. Average percent reduction in physicochemical characteristics of FVW influent in biomethanation process.

The pH of influent was acidic and that of effluent was slightly alkaline. The COD and BOD have 70.37% and 70.31% average reduction from influent to effluent. The average reduction of N, P and TOC is 82.68 %, 89.86% and 79.69% respectively (Table No X).

4. DISCUSSION

The gradual increase in biogas production was observed and similar trend was reported in previous studies irrespective of digester type and feed composition.[14,16-21] The biogas production decreased with decreasing proportion of CDS and BDE and similar observations were reported.[15-17,22] The present study and previous studies[1,3,23] suggest that the HRT is function of pH, feed composition, temperature and type of biogas digester. The optimum level of pH is around neutral or slightly alkaline in most of the studies including the present one.[3,15,19]

5. CONCLUSION

The present study indicates potential of FVW for generation of biogas and methane. It was found that at the optimum pH of slightly alkaline range (7.5) and optimum HRT of 35 days. The volume of biogas generated per liter of FVW slurry (40% solids) was 16-17 litres. The bluish flame in the burning test indicated more than 50% of methane in the biogas.

6. ACKNOWLEDGEMENTS

We are thankful to Principal of Fergusson College and Department of Environmental Science Fergusson College Pune for providing facility for research work. We are also thankful to Pune Municipal Corporation for their help. We are also thankful to management of H.V Desai College Pune for their support and encouragement.

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