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Control of amphibious weed ipomoea (*Ipomoea carnea*) by utilizing it for the extraction of volatile fatty acids as energy precursors

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ABSTRACT

Volatile fatty acids (VFAs), comprising mainly of acetic acid and lesser quantities of propionic and butyric acids, are generated when zoomass or phytomass is acted upon by acidogenic and acetogenic microorganisms. VFAs can be utilized by methanogens under anaerobic conditions to generate flammable methane–carbon dioxide mixtures known as 'biogas'. Acting on the premise that this manner of VFA utilization for generating relatively clean energy can be easily accomplished in a controlled fashion in conventional biogas plants as well as higher-rate anaerobic digesters, we have carried out studies aimed to generate VFAs from the pernicious weed ipomoea (*Ipomoea carnea*). The VFA extraction was accomplished by a simple yet effective technology, appropriate for use even by laypersons. For this acid-phase reactors were set, to which measured quantities of ipomoea leaves were charged along with water inoculated with cow dung. The reactors were stirred intermittently. It was found that VFA production started within hours of the mixing of the reactants and peaked by the 10^{th} or 11^{th} day in all the reactors, effecting a conversion of over 10% of the biomass into VFAs. The reactor performance had good reproducibility and the process appeared easily controllable, frugal and robust.

Introduction

Ipomoea (*Ipomoea carnea,* also called *I. fistulosa*) is among the most dominant and harmful of the weeds that have infested the world's tropical and sub-tropical regions [1,2]. It is an evergreen, flowering, shrub with height ranging from 1.1 to 3 m and stem diameter between 1.5 and 6 cm. It was initially used to make fences but has become very widespread owing to its hardiness, high reproductive success, and very fast rate of growth [3,4]. Its rampant colonization of landmasses and shallow wetlands has proved disastrous in terms of loss of biodiversity, loss of nutrients, and other forms of ecodegradation [5–7].

The weed is so hardy and resilient that it is able to successfully resist all attempts to control it by chemical weedicides or biological agents [8]. Finding a means by which ipomoea can be gainfully utilized appears to be the only way by which it

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can become profitable to regularly harvest the weed, thereby keeping it under some control. Toward this objective efforts have been made to utilize ipomoea as a source of paper pulp [9], biosorbents [2], chemicals [10–12], drugs [13–15], and latex [16]. However, none of these efforts have been economically viable or have shown any potential for large-scale utilization.

About 70% of the biomass contained in ipomoea is due to its leaves and flowers. In the past attempts have been made to utilize these parts of ipomoea as a possible feedstock for generating flammable biogas in anaerobic digesters; for example [17] admixed ipomoea with distillery waste-water to make feedstock for anaerobic digestion. Ipomoea does yield biogas upon anaerobic fermentation [18,19] but no anaerobic digester can be sustainably operated if fed with ipomoea (or any other weed) even in chopped or crushed form because of the following reasons:

(a) Ipomoea cannot be fed to the conventional fixed-dome and floating-dome biogas digesters, of the type which are extensively used in most of the third world countries [20–22] to generate biogas from animal dung-water slurry. This is because the weed does not flow out of the digester exit along with water, as the animal dung-water slurry does, but, instead, accumulates in the digester to eventually clog it. Even when fed as partial feed supplement along with animal dung slurry, the weed eventually clogs the digesters [23–25].
(b) Shredding or mincing of the weed prior to charging does not help either; it makes feeding easy but also leads to equally quick formation of scum which badly clogs the digesters. As a result the digesters become non-functional a few weeks after start-up [25]. In a like manner ipomoea also clogs the continuously stirred tank reactors (CSTR) used in most developed countries for anaerobically digesting piggery and dairy wastes.

But, we reason, if volatile fatty acids (VFAs) can be extracted from ipomoea leaves in the form of aqueous slurry, by acid-phase digestion of the weed, such a slurry can be used as feed for any and all types of anaerobic digesters, low-rate as well as high-rate [26]. In this manner it appears possible to generate clean energy in the form of flammable biogas from about 70% of the biomass contained in ipomoea without jeopardizing any anaerobic digester. The present work has resulted from the pursuit of this strategy. The acid-phase digestion was accomplished in simple, intermittently stirred, tank reactors. The microorganisms required for this purpose were obtained from cow manure, commonly called cow dung, which is rich in the cellulolytic, acidogenic, and acetogenic bacteria, besides enzymes, that are capable of biodegrading phytomass. As rumens are capable of digesting lignocellulosic biomass, their excrement is rich in microorganisms that accomplish the digestion.

Material and methods

All chemicals were analytical regent grade unless otherwise specified. Alkali-resistant glassware and deionized, double-distilled, water were used for all analytical work.

Healthy, adult, plants of ipomoea were collected from locations in and near the Pondicherry University campus. Their leaves were plucked and were liberally washed with water and wiped. Dry weight of the leaves was determined by taking three separate randomly picked samples, weighting them (fresh weight), and then oven drying them at 105 °C to a constant weight. Fresh cow dung, used as inoculum, was obtained from a nearby dairy. Its dry weight was also determined at 105 °C. All the calculations of the VFA yield have been done by taking the dry weight of ipomoea as the basis.

The reactors for VFA extraction consisted of 15 L plastic containers provided with a tap at the bottom to drain off the contents at the end of each experiment. A set of six such reactors were employed, charged as follows:

R_{1A} : Ipomoea 1.5 kg + 12 L water containing 1% cow dung
R_{2A} : As above but without ipomoea
R_{3A} : Ipomoea 1.5 kg + 12 L water containing 2.5% cow dung
R_{4A} : As above but without ipomoea
R_{5A} : Ipomoea 1.5 kg + 12 L water containing 5% cow dung
R_{6A} : As above but without ipomoea

The reactor contents were mixed manually with a fiber–glass rod once every 8 h and the reactor tops were covered with nylon mesh to keep off insects while at the same time ensuring sufficient supply of air to the reactants so that anaerobic conditions do not set in.

Twenty-four hours from the start of each reactor, the contents were stirred and coarse solids were allowed to settle for 10 min. Four 25 mL samples were then drawn from different

Table 1 A typical set of results-pertaining to series B. Figures in percent represent cow dung inoculum (wt%).

Day of reactor operation	VFA content in control reactors, mg/L X			VFA content in the ipomoea-fed reactors, mg/L Y			VFA generated from ipomoea, mg/L $(Y - X)$		
	1.0%	2.5%	5.0%	1.0%	2.5%	5.0%	1.0%	2.5%	5.0%
5th	31.4	94.3	188.6	1953.8	2388.4	3551.9	1922.4	2294.6	3576.4
6th	31.4	94.3	204.3	2050.0	2671.8	3583.4	2012.9	2577.5	3379.1
7th	47.2	110	188.6	2403.6	2923.3	3803.4	2356.4	2813.3	3614.8
8th	31.4	94.3	188.6	2736.8	3300.5	4023.5	2705.4	3206.2	3834.9
9th	31.2	125.7	220.3	3053.7	3960.6	4829.8	3022.5	3834.7	4589.3
10th	62.9	110	220.3	3583.4	3992	4997.9	3300.4	3882	4777.6
11th	47.2	110	251.5	3347.6	38820	5155.1	3347.6	3772	4903.6
12th	59.04	110	188.6	3136.5	3874.5	4335.8	3077.5	3764.5	4147.2
13th	62.9	92.3	157.2	2767.5	3105.8	3636.3	2704.6	3013.5	3479.1

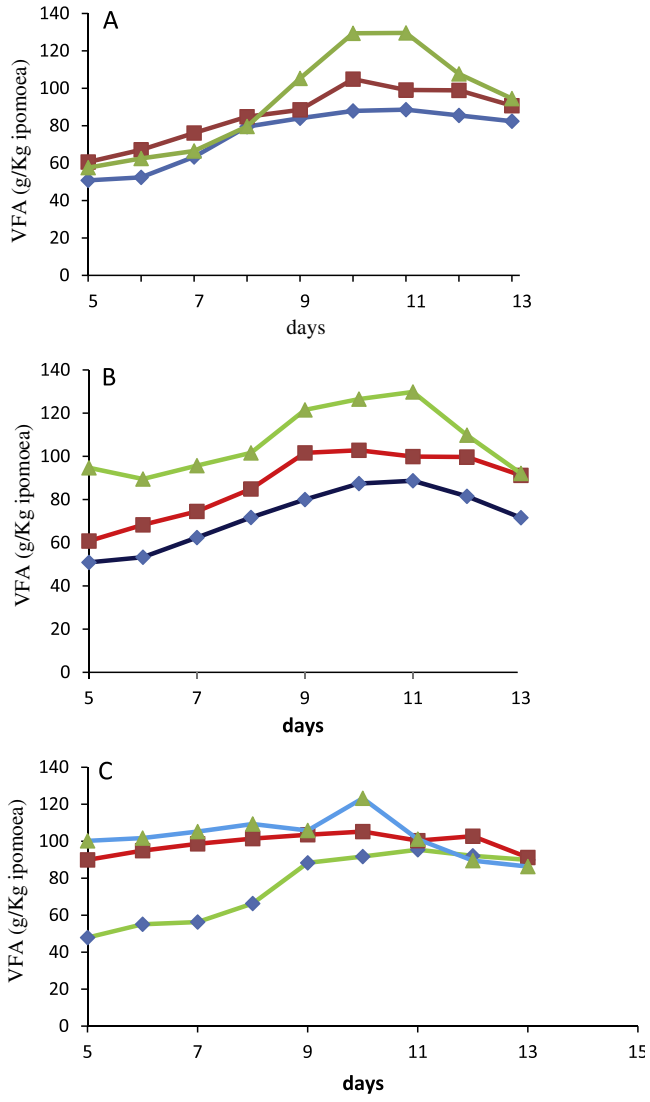

Fig. 1 Pattern of VFA production from ipomoea in the three sets (A, B, C) of reactors inoculated with 1% (◆), 2.5% (■), and 5% (▲) cow dung.

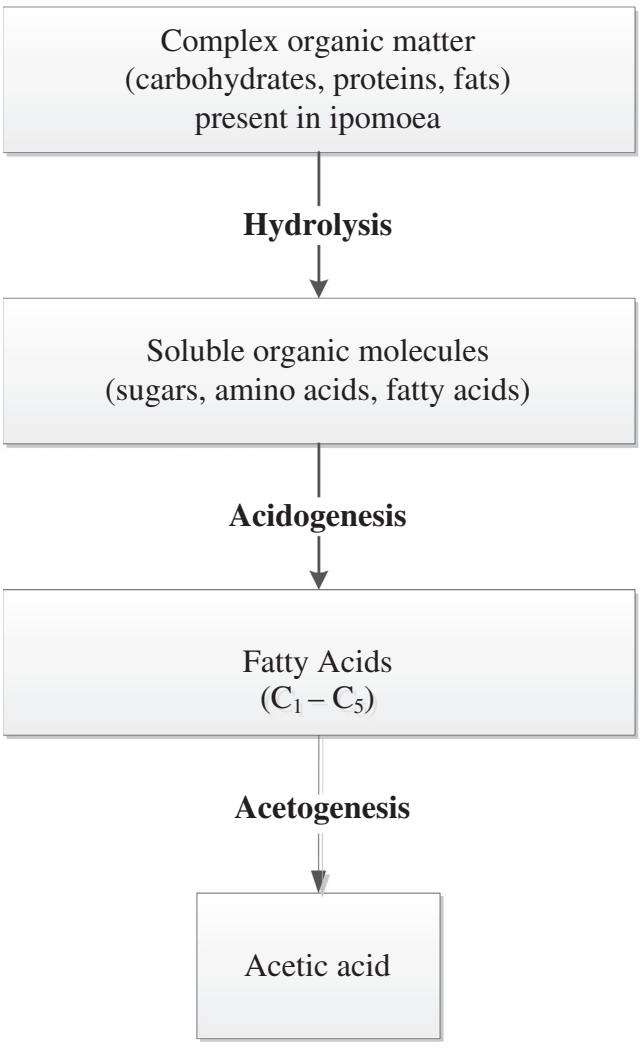

Fig. 2 Steps associated with the production of VFAs from ipomoea.

points in the reactor, and pooled. The volume thus displaced was compensated with an equal volume of water. In subsequent days also, samples were drawn in this manner.

The pooled sample was centrifuged and filtered to remove a few particulates that were present before it was transferred to a 500 mL distillation flask. To it 100 mL of water and 5 mL H_2SO_4 were mixed. After introducing bubblers in the form of the 4–5 pieces of broken glass, the contents were distilled at the rate of about 5 mL per minute. The first 15 mL of distillate was discarded and 150 mL of subsequent distillate was used to estimate VFA concentration by titration with standard NaOH using phenolphthalein indicator. This was in accordance with the distillation-cum-titration procedure described among standard methods [27]. Based on a large number of tests done prior to the analysis of the samples, in which known quantities of acetic acid were distilled and their recoveries quantified, concentration-recovery curves had been obtained for different ranges of acetic acid concentrations. These calibration curves were then used to make the sample VFA assay as accurate as possible. The distillation-cum-titration procedure was preferred by us over the other option [27], which provides for separation by column chromatography and assay by acid–base titration, because the former is quicker, and has adequate accuracy and precision.

After the first round of experiments (series A) was over, the reactors were cleaned and the entire experiment was repeated using a fresh harvest of ipomoea and freshly acquired cow dung (series B). It was done yet again once more (series C). This way reproducibility was tested *vis a vis* VFA extraction carried out with different harvests of ipomoea, different sources of cow dung inoculum, and at different times.

Results and discussion

VFA yield

Tremendous compaction was seen to occur once ipomoea leaves were put under aqueous slurry. Apparently the entrained air which provides the bulk to the leaves is released as the leaves soften under water, leading to agglomeration. Within a few hours the bulk was reduced by several times of

Table 2 Generation of VFA from ipomoea-fed reactors in presence of different concentrations of cow dung (CD) inoculum.

Day of the reactor operation	VFA, g/kg ipomoea, generated in reactors with 1%, CD				VFA g/kg ipomoea, generated in reactors with 2.5% CD				VFA g/kg ipomoea, generated in reactors with 5% CD			
	R_{1A}	R_{1B}	R_{1C}	Average ± SD	R_{3A}	R_{3B}	R_{3C}	Average ± SD	R_{5A}	R_{5B}	R_{5C}	Average ± SD
5th	50.8	50.9	47.9	49.9 ± 1.7	60.6	60.8	89.9	70.4 ± 16.9	67.9	94.7	100.2	87.6 ± 17.3
6th	52.4	53.3	55.1	53.6 ± 1.4	67.1	68.3	94.9	76.8 ± 15.7	72.5	89.5	101.7	87.9 ± 14.7
7th	63.3	62.4	56.3	60.7 ± 3.8	76.1	74.5	98.6	83.1 ± 13.5	76.5	95.7	105.2	92.5 ± 14.6
8th	79.5	71.7	66.3	72.5 ± 6.6	84.8	84.9	101.4	90.4 ± 9.6	89.6	101.6	109.3	100.1 ± 9.9
9th	84	80.0	88.3	84.1 ± 4.2	88.5	101.6	103.5	97.9 ± 8.1	105.3	121.5	105.8	110.9 ± 9.2
10th	87.9	87.4	91.7	89 ± 2.4	104.9	102.8	105.2	104.3 ± 1.3	129.4	126.5	123.2	126.4 ± 3.1
11th	88.6	88.7	95.4	90.9 ± 3.9	99.1	99.9	100.3	99.8 ± 0.6	129.6	129.8	101.1	120.1 ± 16.5
12th	85.5	81.5	92.1	86.4 ± 5.4	98.9	99.7	102.7	100.4 ± 2.0	107.7	109.8	89.5	102.3 ± 11.1
13th	82.4	71.6	89.9	81.3 ± 9.2	90.7	91.2	91.2	91.0 ± 0.3	94.5	92.1	86.3	91.0 ± 4.2

its original volume. The VFA concentration in ipomoea-fed reactors was always 20-times or more than in the control reactors, indicating that VFA production from ipomoea had commenced as soon as the reactors were started. A typical set of results is presented in Table 1. The VFA production, caused by the enzymes and the bacteria present in the cow dung, can be attributed to the first three steps that are known to be associated in the anaerobic digestion of organic substances [28–30] (Fig. 2):

1. The exoenzymes (hydrolase) present in cow dung crack large protein macromolecules, fats, and carbohydrate polymers into water soluble monomers (amino acids, long-chain fatty acids, and sugars).
2. The monomers are then converted into short-chain (C1–C5) fatty acids-principally lactic, propionic, butyric, and valeric acid.
3. The homoacetogenic microorganisms consume these acids to generate acetic acid, carbon dioxide, and hydrogen. Hence the main product, 90% or more, of acetogenesis is acetic acid while minor quantities of propionic acid and traces of higher acids, which had escaped degradation, are also present [31,30].

As may be seen from Table 1, VFA concentrations generated in control reactors have been deducted from VFA concentrations that developed in the ipomoea-fed reactors to obtain VFA generated by the weed alone. From this information VFA generation per kilogram of dried ipomoea has been calculated for all the reactors (Table 2). It may be seen that the relative error in triplicate determinations is mostly less than 10% and is above 15% in only three instances – the 5th and 6th day performances of 2.5% cow dung-inoculated reactors and the 5th day performance of the 5% cow dung-inoculated reactors. During the 10th and 11th day of reactor operation, when VFA levels attained their highest, the relative error was below 5% in five of the six sets. Considering the heterogeneity and natural variability of the reactor feed, and considering the fact that the reactors were operated at different periods of time at ambient temperatures which ranged between 27 °C and 35 °C, the reproducibility in the reactor performance as well as the robustness of the process can be considered as very good.

During the first four days of reactor operation, the VFA yield was low but it approached or crossed 50 g/kg ipomoea by the 5th day. By 13th day the VFA production had passed the peak in all the reactors. Hence the results have been reported for the 5th to 13th day of reactor operation in all cases. The pattern of VFA production in this period in all the three series of experiments is shown in Fig. 1. The VFA yield is seen to peak by the 10th or the 11th day and then declines. In most of the reactors the VFA production followed the order of the cow dung inoculation: 5% > 2.5% > 1% but the difference in peak VFA generation was always less than 20% between successive inoculum concentrations.

All-in-all, VFA yields of the order of 112 ± 12 g per kg of ipomoea were achievable within 10–11 days of reactor operation, representing conversion of over 10% of ipomoea into energy precursors. These precursors can be converted into methane within 24 h or lesser in high-rate anaerobic digesters [26].

Potentially favorable process operation and process economics

The economics of any process essentially depends on the overall hydraulic retention time (HRT) of the process because HRT controls the reactor size which in turn controls the process economics [21]. Of course operational costs are also important but only if they depend on high inputs of energy or cause substantial wastage of materials.

Whereas the continuously stirred tank reactors (CSTRs) which have been tried in the past to process phytomass like ipomoea have an HRT of 15–20 days, and need continuous input of energy for stirring the water-ipomoea slurry, the overall HRT of the presently reported process is under 12 days. Moreover the 10-day acid-phase part requires only occasional stirring hence energy inputs are much lesser.

The VFA-laden slurry is very easy to separate from the parent weed because the latter settles out very quickly. Hence the supernatant of the VFA reactors can be easily transferred to any existing anaerobic digester or to the one specifically set for handling ipomoea-based VFAs. The process has the basic features suitable for scaling up as sequential batch reactors or continuously operated units. It can be said that the process as reported by us is simple, frugal, reproducible, and robust.

Attempts to convert the spent ipomoea into an organic fertilizer by vermicomposting are presently under way so that total disposal of ipomoea can be made possible.

Conclusions

Volatile fatty acids (VFAs) were obtained from the amphibious weed ipomoea (*I. carnea*) in simple to install and easy to operate reactors. The weed was acted upon by the cellulolytic and acidogenic microorganisms present in cow dung with which the reactors were inoculated.

VFA production started within hours of the mixing of the reactants and peaked by the 10^{th} or 11^{th} day in all the reactors, effecting a conversion of over 10% of the biomass into VFAs. As the VFAs are directly utilizable as feed in any and all types of anaerobic digesters to obtain energy in the form of methane, the present work opens up the possibility of large-scale utilization of ipomoea as an energy source.

Conflict of interest

The authors have declared no conflict of interest.

Compliance with Ethics Requirements

This article does not contain any studies with human or animal subjects.

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References

[1] Bhuyan M, Mahanta JJ, Bhattacharyya PR. Biocontrol potential of tortoise beetle (*Aspidomorpha miliaris*) (Coleoptera: Chrysomelidae) on *Ipomoea carnea* in Assam, India. Biocontrol Sci Technol 2008;18(9):941–7.

[2] Sharma A, Bachheti RK. A review on *Ipomoea carnea*. Int J Pharma Bio Sci 2013;4(4):363–77.

[3] Chari KB, Abbasi SA. A study on the fish fauna of oussudu – a rare freshwater lake of south India. Int J Environ Stud 2005;62:137–45.

[4] Konwer D, Kataki R, Saikai M. Production of solid fuel from *Ipomoea carnea* wood. Energy Sources, Part A: Recovery, Utilization Environ Eff 2007;29(9):817–22.

[5] Chari KB, Sharma Richa, Abbasi SA. Comprehensive environmental impact assessment of water resources projects, vol. 1. New Delhi: Discovery Publishing House; 2005, xvi + 580 pages.

[6] Hueza IM, Guerra JL, Haraguchi M, Naoki A, Gorniak SL. The role of alkaloids in *Ipomoea carnea* toxicosis: a study in rats. Exp Toxicol Pathol 2005;57(1):53–8.

[7] Meira M, da Silva EP, David JM, David JP. Review of the genus Ipomoea: traditional uses, chemistry and biological activities. Braz J Pharmacog 2012;22:682–713.

[8] Nandkumar P. Studies on the chemical constituents and the pulp and papermaking characteristics of *Ipomoea Carnea* Jacq. Orient J Chem 2011;27(1):149–54.

[9] Khatiwora E, Adsula VB, Kulkarni M, Deshpande NR, Kashalkar RV. Isolation and characterization of substituted dibutyl phthalate from *Ipomoea carnea* stem. Der Pharma Chem 2013;5(5):5–10.

[10] Ganaie SU, Abbasi T, Anuradha J, Abbbasi SA. Biomimetic synthesis of silver nanoparticles using the amphibious weed ipomoea and their application in pollution control. J King Saud Univ Sci 2014;26(3):222–9.

[11] Anuradha J, Abbasi T, Abbasi SA. Gainful utilization of the highly intransigent weed ipomoea in the synthesis of gold nanoparticles. J King Saud Univ Sci 2014. http://dx.doi.org/10.1016/j.jksus.2014.04.001.

[12] Anuradha J, Abbasi T, Abbasi SA. Use of plants in biomimetic synthesis of gold nanoparticles. J Nano Res; accepted for publication.

[13] Bishayee A, Sarkar A, Chatterjee M. The hepatoprotective activity of carrot (*Daucus carota L*) against carbon tetrachloride intoxication in mouse liver. J Ethnopharmacol 1995;47:69–74.

[14] Gupta RK, Chaudhary S, Singh RK. Antihepatotoxic influence of aqueous extract of *Ipomoea carnea* against carbon tetrachloride induced acute liver toxicity in experimental rodents. Asian J Pharm Clin Res 2012;5(4):262–5.

[15] Rout SK, Kar DM. Sedative, anxiolytic and anticonvulsant effects of different extracts from the leaves of *Ipomoea carnea* in experimental animals. Int J Drug Dev Res 2013;5(2):232–43.

[16] Patel AK, Singh VK, Yadav RP, Moir AG, Jagannadham MV. ICChI, aglycosylated chitinase from the latex of *Ipomoea carnea*. Phytochemistry 2009;70:1210–6.

[17] Deshmukh HV. Economic feasibility and pollution abetment study of biogas production process utilizing admixture of *Ipomoea carnea* and Distillery Waste. J Environ Res Dev 2012;7(2):633–42.

[18] Abbasi SA, Nipaney PC, Schaumberg GD. Bioenergy potential of 8 common aquatic weeds. Biol Waste 1990;34(4):359–66.

[19] Ganesh P, Sanjeevi R, Gajalakshmi S, Ramasamy EV, Abbasi SA. Recovery of methane-rich gas from solid-feed anaerobic digestion of ipomoea (*Ipomoea carnea*). Bioresour Technol 2008;99(4):812–8.

[20] Abbasi T, Abbasi SA. Production of clean energy by anaerobic digestion of phytomass – new prospects for a global warming

amelioration technology. Renew Sustain Energy Rev 2010; 14:1653–9.
[21] Abbasi T, Tauseef SM, Abbasi SA. Anaerobic digestion for global warming control and energy generation: an overview. Renew Sustain Energy Rev 2012;16:3228–42.
[22] Tauseef SM, Premalatha M, Abbasi T, Abbasi SA. Methane capture from livestock manure. J Environ Manage 2013; 117:187–207.
[23] Abbasi SA, Nipaney PC. Generation of biogas from *Salvinia molesta* (Mitchell) on a commercial biogas digester. Environ Technol Lett 1984;5(2):75–80.
[24] Abbasi SA, Nipaney PC. Infestation by aquatic weeds of the fern genus *Salvinia*: its status and control. Environ Conserv 1986;13(3):235–41.
[25] Abbasi SA, Nipaney PC, Ramasamy EV. Studies on multiphase anaerobic-digestion of salvinia. Indian J Technol 1992; 30(10):483–90.
[26] Abbasi T, Tauseef SM, Abbasi SA. Energy recovery from wastewaters with high-rate anaerobic digesters. Renew Sustain Energy Rev 2013;19:704–41.
[27] APHA (American Public Health Association). Standard Methods for the Examination of Water and Wastewater, APHA, Washington, DC, 2012.
[28] Khanal SK. Anaerobic biotechnology for bioenergy production: principles and applications. Wiley-Blackwell; 2008.
[29] Rosenzweig A, Ragsdale SW, editors. Methods in methane metabolism, part a: methanogenesis. Academic Press; 2011.
[30] Abbasi SA, Nipaney PC, Panholzer MB. Biogas production from the aquatic weed pistia (*Pistia Stratiotes*). Bioresour Technol 1991;37(3):211–4.
[31] Abbasi T, Premalatha M, Abbasi SA. Masdar city: a zero carbon zero waste myth. Curr Sci 2012;102, 12.