DEVELOPMENT OF METHANE LOSS MINIMISATION & CARBON DIOXIDE RECOVERY SYSTEMS IN WATER SCRUBBING BASED BIOGAS UPGRADATION

RIMIKA MADAN KAPOOR

CENTRE FOR RURAL DEVELOPMENT & TECHNOLOGY
INDIAN INSTITUTE OF TECHNOLOGY DELHI
HAUZ KHAS, NEW DELHI - 110016

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by

RIMIKA MADAN KAPOOR

Centre for Rural Development & Technology

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CERTIFICATE

The thesis entitled "**Development of Methane Loss Minimisation & Carbon Dioxide Recovery Systems in Water Scrubbing based Biogas Upgradation"** being submitted by **Ms. Rimika Madan Kapoor** to the Indian Institute of Technology Delhi, for the award of the degree of **Doctor of Philosophy**, is a record of bona fide research work carried out by her. She has worked under our supervision and has fulfilled the requirements for the submission of this thesis, which has attained the standard required for a Ph. D. degree of the Institute.

The results presented in this thesis have not been submitted elsewhere for the award of any degree or diploma.

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(Prof. P. M. V. Subbarao)
Professor
Department of Mechanical Engineering
Indian Institute of Technology Delhi,
Hauz Khas, New Delhi – 110016, INDIA

(Prof. Virendra K. Vijay)
Professor
Centre for Rural Development & Technology
Indian Institute of Technology Delhi
Hauz Khas, New Delhi – 110016, INDIA

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ABSTRACT

Biogas, a renewable gas, is a potential alternative to natural gas. It is mostly composed of CH_4 and CO_2 and is obtained during anaerobic fermentation of organic waste. CH_4 makes it a combustible fuel while CO_2 restrains its compressibility and calorific value. To augment its applicability, CO_2 and other impurities like H_2S, moisture are separated from biogas and upgraded to biomethane with above 90% CH_4. Among the variety of biogas cleaning and upgrading methods, water scrubbing is the most feasible method. During the process, raw biogas is split in two major gas streams: the CH_4 rich (above 90%) biomethane stream and the CO_2 rich (80-90%) off stream with significant CH_4 loss being an integral part of the process. Hence, there is an urgent need not only to optimize water scrubbing process to upgrade biogas with above 90% CH_4 but also to develop approaches and sophisticated equipments to maximize recovery of CH_4 loss and separate CO_2 from the off gas stream. In the present study, water scrubbing based biogas upgradation system at IIT Delhi has been investigated in detail. Other major objective of the research are to minimize and recover CH_4 loss and to separate CO_2 with 99.9% purity from the off gas stream of the water scrubbing process.

The performance assessment of water scrubbing based biogas upgradation system revealed various limitations. The water scrubber was redesigned with the help of a MATLAB GUI software and numerous modifications were done in the system to increase its efficiency. Optimisation of the water scrubbing based biogas upgradation system with 150mm and 3000mm scrubbing column diameter and height was accomplished for 95% CH_4 in upgraded biogas. The highest observed CO_2 absorption efficiency of the upgradation system is 97 %. Raw biogas with flow rate of $10Nm^3/h$ at the pressure of 10bar and water flow of $2.0m^3/h$ could be upgraded to 94.8% CH_4 which corresponds to 2.5% CO_2 in upgraded biogas. This is lower than the requirement of 3% CO_2 in biomethane for vehicle utilization in India. The 9.9% CH_4 loss obtained during the process was quite high and needed sophisticated equipment to minimise and thus recover it from the off gas stream of the process.

During the study, it was established that various factors affect and contribute to the CH_4 loss of the system. The flash tank installed in between the water scrubbing column and

desorption tank to partly depressurise water, to minimise CH_4 loss and increase CH_4 recovery has also been studied. It was observed that desorption pressure in the flash tank should be kept low to recover maximum CH_4 from pressurised water coming from the water scrubbing column and retention time of water apt enough to facilitate even small gas bubbles released from water to rise towards the top instead of being dragged into the desorption column. During the study, it was ascertained that the optimum CH_4 recovery of 8.7% was achieved from flash tank at 2 bar pressure and 60 seconds retention time with CH_4 loss% of 0.8% from the desorption tank. The studied flash tank effectively minimised CH_4 loss from 9.9% to 0.8%.

A water scrubber was designed, developed and optimized for CO_2 recovery and it was established that it was capable of retrieving maximum CO_2 from off gas stream of water scrubbing based biogas upgradation plant. At 5Nm^3/h gas flow rate, 5 bar pressure, 1.8m^3/h water flow rate and 2000mm height of the packed bed, the system was efficient to produce 99.9% pure CO_2 at 88.2% CO_2 adsorption efficiency from off gas stream of water scrubbing based biogas upgradation plant. The developed CO_2 recovery system enhanced the efficacy of small-scale biogas upgradation plants by making the complete system carbon negative and cost effective.

The energy required for 10 Nm^3/h capacity water scrubbing based biogas upgradation, compression and bottling plant is 0.575 kWh / Nm^3 of raw biogas.The energy required for 5 Nm^3/h capacity water scrubbing based CO_2 purification system alone is 0.12 kWh / Nm^3 of raw biogas. If both the systems are run simultaneously then the total energy requirement is 0.695 kWh / Nm^3 of raw biogas. Cost Economics of 200 Nm^3/day biogas production, 10Nm^3/h biogas upgradation and bottling plant and 5Nm^3/h CO_2 recovery setup presents that it is a feasible project with payback period of 4 yrs. and high internal rate of interest of 28.58%. The return on investment on the project is high at 46.5% and can be recommended for small scale biogas upgradation and CO_2 recovery setups.

सार

जैव गैस, अक्षयगैस प्राकृषिक, गैस का एक सम्भावित विकल्प है। यह जैविक अपविष्ट के अवायवीय किण्वन के दौरान प्राप्त होता है। जिसमें अधिकतम मात्रा में मेथेन (CH_4) कार्बनडाई आक्साइड (CO_2) होता है। मेथेन (CH_4) एक ज्वलन"ाील ईंधन बनाता है। जबकि कार्बन डाई आक्साइड (CO_2) उसके दबाव व कैलोरीमान को नियंत्रित करता है। इसकी प्रयोज्यता को बढावा देने के लिए कार्बनडाई ऑक्साइड और हाइड्रोजन सल्फाइड तथा नमी आदि को जैव गैस से अलग कर इसे नीवीनकृत किया जाता है। बायो मेथेन जिसमें कि 90 प्रति"ात से ज्यादा मेथेन होता है। बायोगैस सफाई और नवीनीकृत विधियों में जल स्क्रबिंग सबसे व्यवहारिक विधि है। इस प्रक्रिया के दौरान अपरिपक्व जैव गैस दो प्रमुख गैस धाराओं में विभाजित हो जाता हैं। 90 प्रति"ात से ज्यादा CH_4 युक्त मेथेन धारा और (80–90) प्रति"ात कार्बन डाई आक्साइड अधिकता की बन्द धारा जिसमें कुछ उचित मात्रा में मेथेन की कमी उस प्रक्रिया में अन्तर्निहित हैं इसलिए यहां नवीनीकृत जैव गैस जल स्क्रबिंग विधि से 90 प्रति"ात से ज्यादा मेथेन प्राप्ति ही प्रमुख आव"यकता नही हैं वल्कि ऐसी विधि विकसित करना और जटिल संयत्र तैयार करना जो मेथेन की अधिकता बढाये और बन्द धारा से कार्बन डाई आक्साइड (CO_2) को अलग करें। वर्तमान अध्ययन में, आई0 आई0 टी0 दिल्ली में जल स्क्रबिंग आधारित जैवगैस नवीनीकृत प्रणाली की विस्तार से जांच की गई। अनुसंधान के दूसरे प्रमुख उद्दे"य जल स्क्रबिंग में कम से कम मेथेन के नुकसान को ठीक करने के लिए और कार्बन डाई ऑक्साइड (99 प्रति"ात) शुद्धता के साथ बन्द गैस से अलग करना है।

जल स्क्रबिंग आधारित जैवगैस नवीनीकृत प्रणाली प्रद"ान के आकलन की सीमाएं है। मैटलैब जी0 यू0 आई0 साफ्टवेयर की मदद से जल

स्क्रबर का नया स्वरूप बनाया गया और इसकी दक्षता को बढाने के लिए कई सं"ाोधन किये गये। जल स्क्रबिंग आधारित जैव गैस नवीनीकृत प्रणाली का अनुकूलन 150 मि0 मी0 व्यास के साथ और 3000 मि0 मी0 ऊचाई, 95 प्रति"ात CH_4 मेथेन नवीनीकृत जैवगैस के लिए किया जाता है। यह देखा गया नवीनीकृत प्रणाली में कार्बन डाई ऑक्साइड अव"ाोषण दक्षता सबसे ज्यादा 97 प्रति"ात हैं। अपरिपक्व जैव गैस की प्रवाह दर 10 न्यूटन मीटर^3 प्रति घण्टा, 10 बार के दबाव पर और पानी की प्रवाह दर 2 मीटर^3 प्रति घण्टा 94.8 प्रति"ात मेथेन के लिए बढाया गया जो नवीनीकृत जैवगैस में 2.5 प्रति"ात कार्बन डाई ऑक्साइड से मेल खाती हैं। भारत में वाहन उपयोग के लिए जैवगैस मेथेन में 3 प्रति"ात कार्बन डाई ऑक्साइड की आव"यकता से कम हैं 9.9 प्रति"ात मेथेन की कमी इस प्रणाली के दौरान बहुत ज्यादा पायी गई और इसे कम करने के लिए जटिल संयन्त्र की आव"यकता है। जो मेथेन हानि को कम करके बन्द जैव गैस प्रक्रिया से प्राप्त किया जाता है। अध्ययन के दौरान यह पाया गया है कि इस प्रणाली में मेथेन की कमी को विभिन्न तथ्य प्रभावित करते है। मेथेन हानि को कम करने और मेथेन की अधिकतम प्राप्ति के लिए फ्लै"ा टैक को जल स्क्रबिंग स्तभ और डिजार्पसन टैंक के बीच लगाया गया। यह देखा गया है कि जल स्क्रबिंग स्तम्भ से आने वाली दवाबयुक्त जल से अधिकतम मेथेन प्राप्त करने के लिए फ्लै"ा टैंक में डिजार्पसन दबाव कम होना चाहिए और उपयुक्त जल के अवधारण समय को पर्याप्त बनाने के लिए पानी से बने छोटे गैस के बुलबुले को डिजार्पसन स्तम्भ में डालने के बजाय ऊपर उठाना अधिक सुविधा जनक है। अध्ययन के दौरान यह निर्धारित किया गया कि डिजार्पसन टैंक से 0.8 प्रति"ात मेथेन की हानि के साथ 2 बार दबाव और 60 सेकेण्ड तक रखने पर 8.7 प्रति"ात उपयुक्त मेथेन की मात्रा प्राप्त होती है। उपयुक्त फ्लै"ा टैक में मेथेन की हानि को 9.9 प्रति"ात से 0.8 प्रति"ात तक कम किया जा सकता है।

उपयुक्त कार्बन डाई ऑक्साइड की पुनः प्राप्ति के लिए एक जल स्क्रबर निर्मित और विकसित किया गया है। जल स्क्रबिंग बन्द गैस धारा से

अधिकतम कार्बन डाई ऑक्साइड प्राप्त करने में सक्षम हैं। जो जैव गैस उन्नयन संयन्त्र पर आधारित हैं। जब स्क्रबिंग बन्द गैस धारा से 5 न्यूटन मीटर^3 प्रति घण्टा की गैस गवाह दर, 5 बार दबाव, 1.8 मीटर^3 प्रति घण्टा जल प्रवाह दर और 2000 मि0 मी0 ऊचाई के बन्द आधार संयत्र से 88.2 प्रति”ात कार्बन डाई ऑक्साइड डिजार्पसन दक्षता पर 99.9 प्रति”ात शुद्ध कार्बन डाई ऑक्साइड उत्पन्न की जा सकती है। जो कि जैव गैस उन्नयन संयत्र पर आधारित है। कार्बन हानि और उपयुक्त कीमत दर से विकसित पुन कार्बन डाई ऑक्साइड प्राप्ति की प्रणाली द्वारा लघु जैव उन्नयन संयत्र की क्षमता बढाई जा सकती है।

10 न्यूटन मीटर^3 प्रति घण्टा क्षमता वाले जल स्क्रबिंग आधारित जैव गैस उन्नयन, संपीडन और बोतल संयन्त्र के लिए अपरिपक्व जैव गैस की 0.575 किलो वाट प्रति न्यूटन मीटर^3 ऊर्जा आव”यक हैं। 5 न्यूटन मीटर^3 प्रति घण्टा क्षमता वाले कार्बन डाई ऑक्साइड शुद्धधीकरण प्रक्रिया पर आधारित जल स्क्रबिंग संयन्त्र के लिए अपरिपक्व जैव गैस की 0.12 कि0 वाट प्रति न्यूटन मीटर^3 ऊर्जा आव”यक हैं। यदि दोनो प्रणाली एक साथ क्रियान्वित होती है तो अपरिपक्व जैव गैस की 0.695 किलो वाट प्रति न्यूटन मीटर^3 ऊर्जा की आव”यकता होती हैं। आर्थिक दृष्टि से 200 न्यूटन मीटर^3 प्रतिदिन जैव गैस उत्पादन, 10 न्यूटन मीटर^3 प्रति घण्टा जैव गैस उन्नयन और बोतल संयन्त्र तथा 5 मीटर^3 प्रतिघण्टा कार्बन डाई आक्साइड प्राप्ति संरचना 4 वर्ष की निवे”ा वापसी अवधि और 28.58 प्रति”ात ब्याज की उच्च्य आन्तरिक ब्याज दर उपयुक्त परियोजना को प्रदर्”ात करती हैं। इस परियोजना में निवे”ा वापिसी 46.5 प्रति”ात अधिक हैं छोटे पैमाने पर उन्नयन जैव गैस और कार्बन डाई आक्साइड प्राप्ति संयन्त्र के लिए स्वीकार्य है।

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SYMBOLS AND ABBREVIATIONS

%	=	Percent
/	=	Per
<	=	Lower than
>	=	Greater than
^{0}	=	Degree
ε	=	Porosity
μ	=	Chemical potential
ϕ	=	Fugacity
ρ	=	Density
&	=	And
lnϒ	=	Activity coefficient
^{0}C	=	Degree celsius
° F	=	Degree farenhite
Atm	=	Atmospheric
BEP	=	Breakeven point
BV	=	Ball valve
C	=	Carbon
CCS	=	Carbon capture and storage
CH_4% (v/v)UpG	=	CH_4 percentage volume in upgraded biogas
CH_4 (rec) up%	=	CH_4 recovery (upgraded gas)%
CO_2 Ab %	=	CO_2 absorption efficiency
CH_4 loss%	=	CH_4 loss%
CH_4 %(v/v) rg	=	CH_4 volume percentage in released gas from FT / IFT
CO_2 %(v/v) rg	=	CO_2 volume percentage in released gas from FT / IFT

CH_4 (rg)	=	CH_4 volume in released gas from FT / IFT
CH_4 (rec) % rg	=	CH_4 volume percentage in the released gas from FT/IFT
CO_2%(v/v) DeG	=	CO_2 volume percentage in off gas from desorption tank
CO_2 (rec)%	=	CO_2 recovery % from desorption tank
CO_2 (dg)	=	CO_2 volume in off gas from desorption tank
C/N	=	Carbon - nitrogen ratio
CD	=	Cattle dung
CH_4	=	Methane
cm	=	Centimeter
CNG	=	Compressed natural gas
CO	=	Carbon monoxide
CO_2	=	Carbon dioxide
Conc.	=	Concentration
d	=	Day
D	=	Diameter of packed bed column
Dp	=	Diameter of packing
DT1	=	Desorption tank 1
DT2	=	Desorption tank 1
Eq	=	Equivalent
EUDC	=	Extra urban driving cycle
Exp	=	Experimental
EOS	=	Equation of state
Fp	=	Packing factor
FS	=	Full scale
FT	=	Flash tank
g	=	Gram

G	=	Gas flow rate on solute free basis, (kmol/h.m^2)
GC	=	Gas compressor
GFR	=	Gas flow rate
GHG	=	Greenhouse gas
GWP	=	Global warming potential
GJ	=	Giga joule
GI	=	Galvanized iron
GS	=	Gas sample
Gt	=	Giga tones
GUI	=	Graphical user interface
h	=	Hour
hp	=	Horse power
H	=	Hydrogen
H_2O	=	Water
H_2S	=	Hydrogen sulphide
HC	=	Hydro carbon
HPC	=	High pressure compressor
HRT	=	Hydraulic retention time
HTU, H_{tG}	=	Height of transfer units
IFT	=	Intermediate flash tank
IMTP	=	Intelox metal tower packing
IRR	=	Internal rate of return
K	=	Kelvin temperature
K	=	Potassium
K	=	Overall mass transfer coefficient
k	=	Mass transfer coefficient

kg	=	Kilo gram
kJ	=	Kilo joule
km	=	Kilo metre
KVIC	=	Khadi & village industries commission
kW	=	Kilo watt
kWh	=	Kilo watt hour
L	=	Litre
LCBV	=	Level control ball valve
Ls	=	Water flow rate on solute free basis (kmol/h.m^2)
L/G	=	Liquid to gas ratio
LNG	=	Liquefied natural gas
LPG	=	Liquefied petroleum gas
m^3	=	Cubic metre
mg	=	Milli gram
Mat P	=	Material of packing
Min	=	Minute
MJ	=	Mega joule
mL	=	Milli litre
ML	=	Million litre
mm	=	Milli metre
M	=	Molality
MPa	=	Mega pascal
MNRE	=	Ministry of New & Renewable Energy
MPa	=	Mega pascal
MS	=	Mild steel
Mt	=	Million tonne
Mod	=	Modelling work

Mol	=	Mole
MU	=	Mockup
MW	=	Mega watt
N	=	Nitrogen
N_A	=	Rate of transfer
N	=	Normal
NTU, N_{tG}	=	Number of transfer units
O_2	=	Oxygen
OLR	=	Organic loading rate
P	=	Phosphorus
PP	=	Payback period
PS	=	Pilot scale
PCS	=	Pressure control system
PNG	=	Piped natural gas
PR	=	Pressure regulator
ppm	=	Part per million
PVC	=	Poly vinyl chloride
R	=	Gas constant
ROI	=	Return on investment
RT	=	Retention time
rpm	=	Revolution per minute
S_{CG}	=	Schmidt number
SS	=	Stainless steel
STP	=	Standard temperature and pressure
TS	=	Total solids
UDC	=	Urban driving cycle
V	=	Volume

VFA	=	Volatile fatty acids
VS	=	Volatile solids
v/v	=	Volume by volume
WFR	=	Water flow rate
WP	=	Water pump
WS1	=	Water scrubbing column 1
WS2	=	Water scrubbing column 2
w/w	=	Weight by weight
x	=	Mole fraction in liquid phase
X	=	Molar ratio in liquid phase
y	=	Mole fraction in gas phase
Y	=	Molar ratio in gas phase
yr.	=	Year
Z	=	Compressibility factor
Z	=	Height of packed bed
ZMS	=	Zoelite molecular seive