

**EVALUATING THE SUSTAINABILITY OF HYDROTHERMAL
PRETREATMENT AND ANAEROBIC CO-DIGESTION FOR
EXTRACTING BIOENERGY FROM RICE CROP RESIDUES
AND NATURALLY GROWN ALGAL BIOMASS**

SUBODH KUMAR



**CENTRE FOR RURAL DEVELOPMENT &
TECHNOLOGY
INDIAN INSTITUTE OF TECHNOLOGY DELHI**

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by

SUBODH KUMAR

Centre for Rural Development and Technology

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CERTIFICATE

This is to certify that the thesis entitled “**Evaluating the sustainability of hydrothermal pretreatment and anaerobic co-digestion for extracting bioenergy from rice crop residues and naturally grown algal biomass**” being submitted by **Mr. Subodh Kumar** to the Indian Institute of Technology Delhi for the award of the degree of **Doctor of Philosophy** is a record of bonafide research work carried out by him. He has worked under our guidance and supervision and has fulfilled the requirements for the submission of this thesis. To the best of our knowledge, the results contained in this thesis have not been submitted in part or full to any other university or institute for the award of any degree or diploma.



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Prof. Virendra K Vijay

Professor

Centre for Rural Development and Technology

Indian Institute of Technology Delhi

Hauz Khas, New Delhi–110016, India



20/05/2025

Prof. Anushree Malik

Professor

Centre for Rural Development and Technology

Indian Institute of Technology Delhi

Hauz Khas, New Delhi–110016, India

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ABSTRACT

Rice crop residues, including rice straw (RS) and rice husk (RH), possess substantial bioenergy potential to meet a significant portion of energy demand. However, their recalcitrant lignocellulosic structure and imbalanced carbon-to-nitrogen (C/N) ratio pose challenges for converting their bioenergy potential into biogas and biomethane via anaerobic digestion (AD) process. The present study investigated different approaches to overcome these challenges, focusing on improving the biogas and biomethane production from RS and RH by integrating hydrothermal pretreatment and anaerobic co-digestion (AcoD).

This study employed a systematic approach to optimize the process parameters for hydrothermal pretreatment and AD through laboratory-scale batch and continuous feed reactors under controlled conditions. The optimal parameters identified were further tested in a pilot-scale reactor of 300 L under continuous feed mode (using tap water and liquid digestate) over 120 days under ambient conditions. Finally, the feasibility of scaling up the optimized process parameters to an industrial scale was assessed through comprehensive mass balance, energy balance, and environmental impact assessment.

The study was commenced by evaluating the impact of hydrothermal pretreatment on a complex lignocellulosic matrix of RS and RH and their solubilization. In this investigation, pretreatment of RH at 200-250 °C, for 10 min. with total solids (TS) loading of 20%, exhibited significant solubilization of RH. Nevertheless, RS showed more effective solubilization at a lower temperature of 200°C with the same pretreatment conditions. Further, to address the challenge of substantial freshwater input for adjusting high TS (~ 90%) of RS to an optimal range of 20% and the use of NaOH to neutralize its post-pretreatment acidic pH, a novel approach of co-hydrothermal pretreatment of RS and AB was introduced. Interestingly, co-hydrothermal pretreatment of AB and RS at mixing ratios of 9:1, 7:3, and 5:5 resulted in pH values within the alkaline to neutral range. Additionally, the high moisture content of AB eliminated the need for external freshwater to adjust the high TS of RS in the optimum range.

In the batch scale AD experiments, the enhanced solubilization of RH through hydrothermal pretreatment significantly increased the biogas and biomethane yields of RH, resulting in a 4.55-fold and 5.66-fold increase in biogas and biomethane yields, respectively. The highest biogas and biomethane yield of 355 ± 34 mL/g VS_{input} and 153 ± 20 mL/g VS_{input} were achieved for RH pretreated at 230 °C (RH230). Interestingly, RS pretreated at 200 °C (RS200) exhibited almost equivalent biogas and biomethane yields of 373 ± 61 mL/g VS_{input} and 152 ± 28 mL/g VS_{input} , representing 36.63% and 27.73% improvement in biogas and biomethane yield of RS. In contrast,

co-digestion of RS200 with AB at C/N 30 led to a significantly higher increase in biogas and biomethane yield of RS, with improvements of 151.28% and 164.70%, respectively. Additionally, co-hydrothermal pretreatment of AB and RS at a mixing ratio of 5:5 resulted in an improvement of 164.10% and 214.28% in biogas and biomethane yield of RS, respectively, demonstrating the highest biogas and biomethane yield of 721 ± 44 mL/g VS_{input} and 374 ± 25 mL/g VS_{input} , respectively. In the further investigation of optimizing VS loading in continuous digesters of 20 L, the highest specific biogas and biomethane yields were observed at 5 g VS/L in both conditions, including AcoD of RS200 with AB at C/N 30 and AcoD of co-hydrothermally pretreated AB:RS. In the pilot scale investigations conducted in 300 L continuous digesters, AcoD of RH230 with AB at C/N 30 exhibited 17.68% more biomethane yield (193 L/kg VS_{input}) compared to AD of RH230 (164 L/kg VS_{input}), indicating that integration of hydrothermal pretreatment and AcoD is also effective for improving the biogas and biomethane yield for RH. However, the AcoD of RS200 with AB at C/N 30 exhibited a comparatively higher biomethane yield (303 L/kg VS_{input}) in similar pilot-scale experiments. Using liquid digestate instead of tap water in digester feeding was ineffective as it reduced the biogas and biomethane yield in all pilot-scale studies.

In the theoretical assessment on the feasibility of scaling up the lab-based findings to an industrial scale, the AcoD of RH230 with AB at C/N 30 was more promising than the AD of RH230. This is due to its higher mass conversion into biogas, higher biomethane yield, higher net energy output, higher energy recovery, and minimal GHG emissions. In contrast, the AcoD of RS200 with AB at C/N 30 and the AcoD of co-hydrothermally pretreated AB and RS (mixed at the ratio 5:5) exhibited nearly similar biomethane yield, net energy output, and GHG emissions. AcoD of co-hydrothermally pretreated AB and RS exhibited higher mass conversion into biogas. However, higher energy recovery in the AcoD of RS200 with AB at C/N 30 makes it the best strategy for sustainable biogas and biomethane production from RS. The theoretical analysis conducted on the use of the liquid fraction of digestate and CO₂-rich pressurized water as nutrient sources for AB cultivation indicated a reduction in overall GHG emissions, with the highest reduction for AcoD of co-hydrothermally pretreated AB and RS. Overall, integrating hydrothermal pretreatment with co-digestion was noted to be an energy-efficient, environmentally friendly, and sustainable strategy for biomethane production from RS, RH, and AB.

Future studies are recommended to focus on integrating solar thermal heating and nutrient recovery from liquid digestate through AB cultivation and evaluating the effects of various nutrients present in solid digestate on crop productivity. Additionally, performing sensitivity analyses to study the impact of fluctuations in critical parameters on overall performance is also advised.

सार

धान के फसल अवशेषों, जिनमें धान का पुआल (rice straw) और धान की भूसी (rice husk) शामिल हैं, में ऊर्जा उत्पादन की पर्याप्त संभावनाएं हैं, जो ऊर्जा की मांग के एक महत्वपूर्ण हिस्से को पूरा कर सकती हैं। हालांकि, इनकी जटिल लिग्नोसेलूलोसिक संरचना और असंतुलित कार्बन-नाइट्रोजन (C/N) अनुपात, इनकी जैव ऊर्जा क्षमता को बायोगैस और बायोमीथेन में बदलने की प्रक्रिया में चुनौतियाँ उत्पन्न करते हैं। इस अध्ययन में इन चुनौतियों को दूर करने के लिए विभिन्न दृष्टिकोणों की जांच की गई, जिसमें धान का पुआल और धान की भूसी से बायोगैस और बायोमीथेन उत्पादन को हाइड्रोथर्मल प्रीट्रीटमेंट और सह-अवायवीय पाचन (anaerobic co-digestion) के एकीकरण द्वारा सुधारने पर ध्यान केंद्रित किया गया।

इस अध्ययन ने हाइड्रोथर्मल प्रीट्रीटमेंट और अवायवीय पाचन (anaerobic digestion) के प्रक्रिया मापदंडों (process parameters) का अनुकूलन (optimization) प्रयोगशाला आधारित बैच और सतत फीड रिएक्टरों में एक व्यवस्थित पद्धति के अनुसार नियंत्रित परिस्थितियों में किया गया। प्रयोगशाला आधारित प्रयोगों से प्राप्त हुए अनुकूलतम मापदंडों का परीक्षण 300 लीटर क्षमता वाले पायलट स्केल बायोगैस रिएक्टर में निरंतर फीड मोड में (नल के पानी और डाइजेस्ट के तरल अंश का उपयोग करके) 120 दिनों के दौरान परिवेशी परिस्थितियों (ambient conditions) में किया गया। अंततः, अनुकूलित प्रक्रिया मापदंडों को औद्योगिक पैमाने पर बढ़ाने की संभावना का आकलन व्यापक द्रव्यमान संतुलन, ऊर्जा संतुलन और पर्यावरणीय प्रभाव मूल्यांकन के माध्यम से किया गया।

अध्ययन की शुरुआत धान के पुआल और धान की भूसी की जटिल लिग्नोसेलूलोसिक संरचना को तोड़कर इनकी घुलनशीलता पर हाइड्रोथर्मल प्रीट्रीटमेंट के प्रभाव का मूल्यांकन करके की गई। इस अध्ययन में, धान की भूसी का 20% ठोस सामग्री (TS) लोडिंग के साथ, 200-250°C पर 10 मिनट के लिए हाइड्रोथर्मल प्रीट्रीटमेंट, धान की भूसी की घुलनशीलता बढ़ाने में प्रभावी पाया गया। हालांकि, समान प्रीट्रीटमेंट परिस्थितियों में धान के पुआल ने 200°C के निचले तापमान पर अधिक प्रभावी घुलनशीलता दिखाई। हालांकि धान के पुआल की ठोस सामग्री (TS~90%) को 20% की इष्टतम सीमा में समायोजित करने के लिए बड़ी मात्रा में ताजे पानी की आवश्यकता और प्रीट्रीटमेंट के बाद इसके अम्लीय pH को संतुलित करने के लिए NaOH का उपयोग इसके सतत उपयोग में अभी भी एक बड़ी चुनौती थी जिसे हल करने के लिए धान के पुआल और शैवाल बायोमास (algal biomass) के सह-हाइड्रोथर्मल प्रीट्रीटमेंट का एक नया दृष्टिकोण प्रस्तुत किया गया। दिलचस्प बात यह है कि 9:1, 7:3, और 5:5 के मिश्रण अनुपात में धान के पुआल और शैवाल बायोमास के सह-हाइड्रोथर्मल प्रीट्रीटमेंट में pH का मान क्षारीय से लेकर संतुलित सीमा में प्राप्त हुआ। इसके अलावा, शैवाल बायोमास की उच्च नमी ने धान के पुआल के उच्च ठोस सामग्री (TS~90%) को इष्टतम सीमा में समायोजित करने के लिए बाहरी स्रोत से ताजे पानी की आवश्यकता को समाप्त कर दिया।

बैच स्केल अवायवीय पाचन प्रयोगों में, हाइड्रोथर्मल प्रीट्रीटमेंट के माध्यम से धान की भूसी की बढ़ी हुई घुलनशीलता ने बायोगैस और बायोमीथेन के उत्पादन को क्रमशः 4.55 और 5.66 गुना तक बढ़ा दिया, जिससे 230°C (RH230) पर प्रीट्रीटेड धान की भूसी के लिए क्रमशः 355±34 mL/g VS_{input} और 153±20 mL/g VS_{input} की उच्चतम बायोगैस और बायोमीथेन प्राप्त हुई। दिलचस्प बात यह है कि 200°C पर प्रीट्रीटेड धान के पुआल (RS200) ने क्रमशः 373±61 mL/g VS_{input} और 152±28 mL/g VS_{input} का लगभग समान बायोगैस और बायोमीथेन उत्पादन प्रदर्शित किया, जो धान के पुआल की बायोगैस और बायोमीथेन उत्पादन में क्रमशः 36.63% और 27.73% सुधार को प्रदर्शित करता है। हालांकि, C/N 30 पर शैवाल बायोमास (algal biomass) के साथ 200°C पर प्रीट्रीटेड धान के पुआल (RS200) का सह-अवायवीय पाचन अधिक प्रभावशाली साबित हुआ जो बायोगैस और बायोमीथेन के उत्पादन में क्रमशः 151.28% और 164.70% की महत्वपूर्ण वृद्धि का कारण बना। इसके अलावा, 5:5 के मिश्रण अनुपात में शैवाल बायोमास और धान के पुआल का सह-हाइड्रोथर्मल प्रीट्रीटमेंट ने धान के पुआल के बायोगैस और बायोमीथेन उत्पादन में क्रमशः 164.10% और 214.28% की वृद्धि की, जिससे क्रमशः 721±44 mL/g VS_{input} और 374±25 mL/g VS_{input} की उच्चतम बायोगैस और बायोमीथेन प्राप्त हुई।

20 L क्षमता वाले सतत डाइजेस्टर में वाष्पशील ठोस (volatile solids) लोडिंग को अनुकूलित करने के आगे के अन्वेषण में, C/N 30 पर शैवाल बायोमास के साथ 200°C पर प्रीट्रीटेड धान के पुआल (RS200) का सह-अवायवीय पाचन और सह-हाइड्रोथर्मली प्रीट्रीटेड शैवाल बायोमास और धान के पुआल के सह-अवायवीय पाचन सहित दोनों परिस्थितियों में 5 g VS/L पर उच्चतम विशिष्ट बायोगैस और बायोमीथेन उत्पादन देखा गया।

300 L क्षमता वाले सतत डाइजेस्टर्स में किए गए पायलट स्केल के अन्वेषणों में, C/N 30 पर शैवाल बायोमास के साथ 230°C पर प्रीट्रीटेड धान की भूसी (RH230) के सह-अवायवीय पाचन का, धान की भूसी के एकल-पाचन की तुलना में 17.68% अधिक (193 L/kg VS_{input}) बायोमीथेन का उत्पादन करना यह इंगित करता है कि हाइड्रोथर्मल प्रीट्रीटमेंट और सह-अवायवीय पाचन का एकीकरण धान की भूसी की बायोगैस और बायोमीथेन पैदावार में सुधार के लिए भी प्रभावी है। हालांकि, C/N 30 पर शैवाल बायोमास के साथ 200°C पर प्रीट्रीटेड धान के पुआल (RS200) के सह-अवायवीय पाचन ने अपेक्षाकृत अधिक बायोमीथेन के उत्पादन (303 L/kg VS_{input}) को प्रदर्शित किया। पायलट स्केल अध्ययनों में सभी डाइजेस्टर फीडिंग में नल के पानी के बजाय डाइजेस्ट के तरल अंश का उपयोग अप्रभावी साबित हुआ क्योंकि इससे बायोगैस और बायोमीथेन के उत्पादन में कमी आई।

प्रयोगशाला-आधारित निष्कर्षों को औद्योगिक पैमाने पर बढ़ाने की व्यवहार्यता के सैद्धांतिक मूल्यांकन में पाया गया कि C/N 30 पर शैवाल बायोमास के साथ 230°C पर प्रीट्रीटेड धान की भूसी (RH230) का सह-

अवायवीय पाचन, 230°C पर प्रीट्रीटेड धान की भूसी (RH230) के एकल-अवायवीय पाचन (mono-anaerobic digestion) की तुलना में अधिक प्रभावी है। इसका कारण यह है कि इसमें द्रव्यमान का बायोगैस में उच्च मात्रा में रूपांतरण, अधिक बायोमीथेन उत्पादन, अधिक शुद्ध ऊर्जा आउटपुट, अधिक ऊर्जा पुनर्प्राप्ति और न्यूनतम ग्रीनहाउस गैस (greenhouse gases) का उत्सर्जन होता है। इसके विपरीत, C/N 30 पर शैवाल बायोमास के साथ 200°C पर प्रीट्रीटेड धान के पुआल (RS200) के सह-पाचन और सह-हाइड्रोथर्मल प्रीट्रीटेड शैवाल बायोमास और धान के पुआल (5:5 अनुपात में मिश्रित) का सह-अवायवीय पाचन समान बायोमीथेन उत्पादन, शुद्ध ऊर्जा आउटपुट और ग्रीनहाउस गैस उत्सर्जन प्रदर्शित करते हैं। सह-हाइड्रोथर्मल प्रीट्रीटेड शैवाल बायोमास और धान के पुआल (5:5 अनुपात में मिश्रित) का सह-अवायवीय पाचन अधिक द्रव्यमान रूपांतरण दिखाता है, लेकिन C/N 30 पर शैवाल बायोमास के साथ 200°C पर प्रीट्रीटेड धान के पुआल (RS200) के सह-पाचन में उच्च ऊर्जा पुनर्प्राप्ति इसे धान के पुआल से बायोगैस और बायोमीथेन उत्पादन के लिए सर्वोत्तम रणनीति बनाती है।

डाइजेस्ट के तरल अंश और CO₂-समृद्ध दबावयुक्त पानी का पोषक स्रोत के रूप में शैवाल बायोमास के उत्पादन के लिए उपयोग करने के सैद्धांतिक मूल्यांकन में पाया गया कि इससे कुल ग्रीनहाउस गैस उत्सर्जन में कमी आई, और सबसे अधिक कमी सह-हाइड्रोथर्मल प्रीट्रीटेड शैवाल बायोमास और धान के पुआल का सह-अवायवीय पाचन में हुई। कुल मिलाकर, हाइड्रोथर्मल प्रीट्रीटमेंट और सह-पाचन के एकीकरण को ऊर्जा-कुशल, पर्यावरण-अनुकूल और धान के पुआल, धान की भूसी, और शैवाल बायोमास से बायोमीथेन उत्पादन के लिए एक उचित रणनीति माना गया।

भविष्य के अध्ययनों में सौर थर्मल हीटिंग और डाइजेस्ट के तरल अंश से पोषक तत्वों की पुनर्प्राप्ति को शैवाल बायोमास के उत्पादन के साथ एकीकृत करने और ठोस पाचन में मौजूद विभिन्न पोषक तत्वों का फसल उत्पादन पर प्रभाव का मूल्यांकन करने पर ध्यान केंद्रित करने की सिफारिश की गई है। इसके अलावा, समग्र प्रदर्शन पर महत्वपूर्ण मापदंडों में उतार-चढ़ाव के प्रभाव का अध्ययन करने के लिए संवेदनशीलता विश्लेषण करने की भी सलाह दी गई है।

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SYMBOLS AND ABBREVIATIONS

RS	Rice straw
RH	Rice husk
RB	Rice bran
AB	Algal biomass
AD	Anaerobic digestion
AcoD	Anaerobic co-digestion
TGA	Thermogravimetric analysis
DTA	Derivative thermogravimetric analysis
SEM	Scanning Electron Microscopy
TCOD	Total chemical oxygen demand
sCOD	Soluble chemical oxygen demand
TDS	Total dissolved solids
EC	Electrical conductivity
ORP	Oxidation-reduction potential
VFAs	Volatile fatty acids
WB	Wet basis
VS	Volatile solids
TS	Total solids
FC	Fixed carbon
OLR	Organic loading rate
HRT	Hydraulic retention time
GHG	Greenhouse gases
GWP	Global warming potential
VOC	Volatile organic carbons
C/N	Carbon to nitrogen ratio
S/I	Substrate to inoculum ratio
RSM	Response surface methodology
CNG	Compressed Natural Gas
COD	Chemical oxygen demand
w/v	Weight by volume
ASTM	American Society for Testing and Material
v/v	Volume by volume

ppm	Parts per billion
ppb	Parts per million
ppt	Parts per thousand
MT	Million metric tonnes
min	Minute
kg	Kilogram
g	Gram
MJ	Megajoule
kJ	Kilojoule
L	Liter
H	Hour
D	Day
mm	Millimeter
mL	Milliliter
MWh	Mega-watt hour
>	Greater than
<	Less than
/	Per
µg	Microgram
µM	Micromole
°C	Degree Celsius
H ₂	Hydrogen
CH ₄	Methane
CO ₂	Carbon dioxide
Ca(OH) ₂	Calcium hydroxide
NaOH	Sodium hydroxide
N ₂ O	Nitrous oxide
CO ₂ -eq	Carbon dioxide equivalent
NH ₃	Ammonia
H ₂ S	Hydrogen sulfide
C	Carbon
H	Hydrogen
N	Nitrogen
S	Sulfur

O	Oxygen
P	Phosphorus
K	Potassium
SO ₂	Sulfur dioxide
NO _x	Nitrogen oxides
NaOH	Sodium Hydroxide
HCl	Hydrochloroic acid
HNO ₃	Nitric acid
H ₃ PO ₄	Phosphoric acid
H ₂ SO ₄	Sulfuric acid
CH ₃ COOH	Acetic acid
H ₂ C ₂ O ₄	Oxalic acid
C ₄ H ₄ O ₄	Fumaric acid
Na ₂ CO ₃	Sodium carbonate
H ₂ O ₂	Hydrogen peroxide
Fe	Iron
Ni	Nickle
Mo	Molybdenum
Co	Cobalt
Cu	Copper
W	Tungsten
Se	Selenium
Fe ₃ O ₄	Ferrous Ferric oxide
TiO	Titanium oxide
SiO ₂	Silicon oxide
CaO	Calcium oxide
P ₂ O ₅	Phosphorus oxide
K ₂ O	Potassium oxide
MgO	Magnesium oxide
SO ₃	Sulfur trioxide
Na ₂ O	Sodium oxide
Al ₂ O ₃	Aluminium oxide
Fe ₂ O ₃	Ferric oxide
TiO ₂	Titanium dioxide

MnO	Manganese oxide
Cr ₂ O ₃	Chromic oxide
Cl	Chlorine
ZnO	Zinc oxide
K ₂ Cr ₂ O ₇	Potassium dichromate
SiC	Silicon carbide
Si ₃ N ₄	Silicon Nitride
Mg ₂ Si	Magnesium silicide