ENERGY EFFICIENT BIOHYDROGEN PRODUCTION FROM A SEA WEED (*Ulva reticulate*)

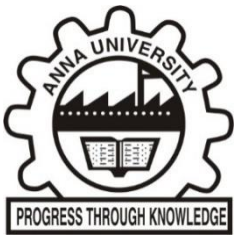
A THESIS

Submitted by

DINESH KUMAR M

in partial fulfillment of the requirements for the degree of

DOCTOR OF PHILOSOPHY



FACULTY OF CIVIL ENGINEERING

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CERTIFICATE

This is to certify that all corrections and suggestions pointed out by the Indian /Foreign Examiner(s) are incorporated in the Thesis titled " ENERGY EFFICIENT BIOHYDROGEN PRODUCTION FROM A SEA WEED (Ulva reticulate) " submitted by Mr. Dinesh Kumar.M

Signature of Research Co-ordinator

Signature of the Supervisor

24/02/2021

Place : Thoothukudi

Date : 24/02/2021

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Proceedings of the Ph.D. Viva-Voce Examination of Mr.Dinesh Kumar.M held at 03:00 PM on 24.02.2021 in EDUSAT HALL (First Floor, F-08), Department of Civil Engineering, University College of Engineering (VOC College of Engineering) Thoothukudi-628008

The Ph.D. Viva-Voce Examination of Mr.Dinesh Kumar.M (Reg. No. 17141891129) on his/her Ph.D. Thesis Entitled " ENERGY EFFICIENT BIOHYDROGEN PRODUCTION FROM A SEA WEED (Ulva reticulate) " was conducted on **24.02.2021** at 03:00 PM in the EDUSAT HALL (First Floor, F-08), Department of Civil Engineering, University College of Engineering (VOC College of Engineering) Thoothukudi-628008.

The following Members of the Oral Examination Board were present:

1.	Dr. P.Sivashanmugam,Professor(HAG),Department of Chemical Engineering,National Institute of Technology ,Tiruchirapalli - 620015.	Indian Examiner
2.	Dr. C.Lajapathi Rai,Senior Principal Scientist,Department of Chemical Engineering,CSIR-Central Leather Research Institute,Adyar Chennai - 600020.	Subject Expert
3	Dr. Rajesh Banu.J,Associate Professor, Department of Life Science, Central University of Tamilnadu,Tiruvarur	Supervisor
4	Dr.S Adish Kumar,Assistant Professor,Department of Civil Engineering,University VOC College of Engineering, Anna University - Thoothukudi Campus, Thoothukudi - 628006.	Research Co-ordinator/ Convener

The research scholar, Mr. Dinesh Kumar.M presented the salient features of his/her Ph.D. work. This was followed by questions from the board members. The questions raised by the Foreign and Indian Examiners were also put to the scholar. The scholar answered the questions to the full satisfaction of the board members.

The corrections suggested by the Indian/Foreign examiner have been carried out and incorporated in the Thesis before the Oral examination.

Based on the scholars research work, his/her presentation and also the clarifications and answers by the scholar to the questions, the board recommends that Mr.Dinesh Kumar.M be awarded Ph.D. degree in the **Faculty of Civil Engineering**.

Indian Examiner

Subject Expert

Research Co-ordinator/
Convener

Supervisor

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BONAFIDE CERTIFICATE

The research work embodied in the present Thesis entitled **"ENERGY EFFICIENT BIOHYDROGEN PRODUCTION FROM A SEA WEED (*Ulva reticulate*)"** has been carried out in the Department of Civil Engineering, Anna University Regional Campus, Tirunelveli. The work reported herein is original and does not form part of any other thesis or dissertation on the basis of which a degree or award was conferred on an earlier occasion or to any other scholar.

I understand the University's policy on plagiarism and declare that the thesis and publications are my own work, except where specifically acknowledged and has not been copied from other sources or been previously submitted for award or assessment.

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ABSTRACT

Marine macroalgae (*Ulva reticulate*) also known as seaweeds are widely available in coastal region and are considered as a potential feedstock for bioenergy production. It gains more attention towards biofuel production due to some advantages such as ease available, high productivity, and high conversion rate. Hydrogen is considered as clean fuel because during combustion water vapour is the only by product with no emission of carbon dioxide. Biohydrogen is used as main alternative fuel source for other fossil fuels and considered as sustainable energy with high energy yield. Biohydrogen production can be performed through anaerobic fermentation which mainly consists of hydrolysis and acidogenesis. Due to the intact algal cell membrane, hydrolysis process becomes slow thus seriously constrains consequent acidogenesis for hydrogen production. Thus, pretreatment is necessary step to disintegrate the algal cell and enhances the hydrogen production.

Various pretreatment such as physical (microwave, thermal), chemical (acid, alkali, and surfactant), mechanical (sonication, dispersion) and biological (enzyme, bacteria) methods are being employed to disintegrate the cell wall, to hydrolyze the complex components into simple substances. Microwave irradiation is effective pretreatment method to disintegrate the algal biomass. Although, it is an effective method and has a limitation of high-power consumption. Therefore, by combining with chemicals, the energy input can be significantly reduced and algal solubilisation can be greatly enhanced simultaneously.

Firstly, microwave pretreatment was done by varying the microwave power (MP) 90 – 630 W (power intensity - 10-70%). Optimum microwave conditions were identified by using the release of organics and

biopolymers (protein and carbohydrate). Maximum organics release of 1450 mg/L was observed at 360 W at 15 min time. Then, at optimum microwave condition, H_2O_2 dosages were introduced by varying the dosages from 0.003 to 0.03 g H_2O_2/g TS and H_2O_2 induced microwave (HMW) pretreatment was carried out. At 0.024 g H_2O_2/g TS, maximum release of organics and solubilisation of 1630 mg/L and 33.9% were achieved.

In first phase, effect of acidic pH on soluble organic release in acidic - H_2O_2 induced microwave (AHMW) were carried out. The experiment was carried out by varying the pH from 4 – 6 at optimized HMW condition. Maximum release of organic (1850 mg/L), carbohydrate (888.3 mg/L) and protein (699.3 mg/L) were observed at pH 5. Biohydrogen assay of pretreated samples in hydrogen yield was carried out and higher biohydrogen generation of 95 mL H_2/g COD was observed in AHMW compared than others.

In second phase, effect of alkaline pH on soluble organics release in alkaline - H_2O_2 induced microwave (ALHMW) were carried out. In this phase, by varying the pH from 8-12 in optimized HMW condition was done. The rapid organic release was identified at 8 min time. At pH 10, 1750 mg/L of maximum organic release was observed. As the result, higher biohydrogen production of 87.5 mL H_2/g COD was achieved in ALHMW compared to other samples.

In third phase, surfactant was introduced in optimized microwave conditions to reduce the energy consumption. In this phase, the experiment was carried out by varying the surfactant dosages from 0.003 – 0.03 g ADS/g TS in optimized MW condition. Maximum organic release of 1600 mg/L was released in 0.024 g ADS/g TS. Maximum biohydrogen generation of 80 mL H_2/g COD was achieved in SMW.

Finally, the comparative investigation of maximum solubilisation efficiency and biohydrogen production during various pretreatment was carried out. Based on energy analysis, acidic - H_2O_2 induced microwave (AHMW) pretreatment yields higher net energy than others.