EVALUATION OF PHASE SEPARATED MICROWAVE DISINTEGRATION OF DAIRY WASTE ACTIVATED BIOMASS FOR METHANE PRODUCTION

A THESIS

Submitted by

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in partial fulfillment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

FACULTY OF CIVIL ENGINEERING

ANNA UNIVERSITY

CHENNAI 600 025

AUGUST 2018

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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 " EVALUATION OF PHASE SEPARATED MICROWAVE DISINTEGRATION OF DAIRY WASTE ACTIVATED BIOMASS FOR METHANE PRODUCTION " submitted by Mr./Ms. Parvathy Eswari.A

Signature of the Supervisor

Place : Tirunelveli

Date : 30/08/2018

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Proceedings of the Ph.D. Viva-Voce Examination of ~~Mr.~~/Ms.Parvathy Eswari.A held at 11:00 AM on 30.08.2018 in Conference Hall, Department of Civil Engineering, Anna University Regional campus Tirunelveli.

The Ph.D. Viva-Voce Examination of ~~Mr.~~/Ms.Parvathy Eswari.A (Reg. No. 1424189701) on ~~his~~/her Ph.D. Thesis Entitled " EVALUATION OF PHASE SEPARATED MICROWAVE DISINTEGRATION OF DAIRY WASTE ACTIVATED BIOMASS FOR METHANE PRODUCTION " was conducted on **30.08.2018** at 11:00 AM in the Conference Hall, Department of Civil Engineering, Anna University Regional campus Tirunelveli..

The following Members of the Oral Examination Board were present:

1.	Dr. K.N.Yogalakshmi,Assistant Professor,School of Environmental & Earth Sciences,Central University of Punjab,Bathinda- 151001	Indian Examiner
2.	Dr. P.Sivashanmugham,Professor,Department of Chemical Engineering,National Institute of Technology Trichy,Tiruchirappalli 620015	Subject Expert
3.	Dr. Rajesh Banu.J,Assistant Professor, Department of Civil Engineering, Regional Centre of Anna University of Anna University,Tirunelveli	Supervisor & Convenor

The research scholar, ~~Mr.~~/Ms. Parvathy Eswari.A 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.~~/Ms.Parvathy Eswari.A be awarded Ph.D. degree in the **Faculty of Civil Engineering**.

30/8/2018

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CERTIFICATE

The research work embodied in the present Thesis entitled **"EVALUATION OF PHASE SEPARATED MICROWAVE DISINTEGRATION OF DAIRY WASTE ACTIVATED BIOMASS FOR METHANE PRODUCTION"** has been carried out in the Department of Civil Engineering, Regional Campus of Anna University, 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

Waste Activated Biomass (WAB) produced from dairy industries is estimated to be 5% to 25% of total volume of treated effluent with high proportion of biodegradable organic matter. To minimize the environmental problems, proper treatment and disposal methods have been adopted, which in turn affects the overall economy of the dairy industry. Biomass stabilization was effectively carried out with Anaerobic Digestion (AD) and also it obtains energy in the form of biomethane. The limitation in AD can be overcome by a number of disintegration processes such as mechanical, thermal, physical, chemical and biological disintegration. Microwave (MW) irradiation is one of the most potent heat treatment processes for WAB. Still this method has some disadvantages such as high costs and energy consumption. Perhaps it can be made profitable through phase separated microwave disintegration of biomass (Extracellular Polymeric Substances (EPS) dissociation followed by biomass disintegration) which leads to higher yield and minimal energy input. Numerous methods can be adopted for dissociating EPS from the microbial cell wall. The chemical methods with the use of chemicals such as H_2O_2, zeolite and EGTA were used in this study.

The study aims to improve the biomethane potential of dairy Waste Activated Biomass (WAB) by H_2O_2 mediated Microwave disintegration (HA-MW) approach. The results of HA-MW compared with the Microwave disintegration (MW) for energy and economic factors. In the phase separated disintegration process, the H_2O_2 concentration of about 0.5 mg/g Suspended Solids (SS) under acid pH of 5 was found to be optimum for effective dissociation of EPS matrix. Higher biomass lysates liquefaction (Chemical Oxygen Demand (COD) solubilization) and solids reduction of about 46.6%

and 35% was achieved HA-MW when compared to that of MW (30% and 24.7%). HA-MW disintegration decreases the amount of specific energy essential to disintegrate the biomass from 18,900 kJ/kg TS to 10,810 kJ/kg TS, when compared to MW. It subsequently improved the methane yield of about 250 mL/g VS in HA-MW, which was 9.6% higher than MW. A net profit of about 3787 INR /Ton of SS was achieved for HA-MW, therefore it is highly recommended for WAB disintegration.

In the next part of the study, an attempt has been made to improve hydrolysis of waste activated biomass by zeolite. It act as a cation binding agent for the effective removal of extra cellular polymeric substances. The outcome of the experiment confirmed that 0.04 g/g SS of zeolite was perceived to be optimal dosage for EPS dissociation with negligible biomass disintegration. The degree of dissociation was in the range of 93%, which reveals that 0.04 g/g SS of zeolite was best for the dissociation of EPS. To evaluate the impact of EPS dissociated biomass disintegration, the biomass was exposed to microwave disintegration and zeolite mediated microwave disintegration. The result of the microwave disintegration shows that the solids reduction and biomass lysates liquefaction was 33.1% and 42.7% comparatively more than microwave disintegrated (24.7% and 30%) sample. Most importantly, zeolite usage reduced microwave specific energy (16,200 kJ/kg TS) applied for liquefaction of biomass. It subsequently improved the methane yield of about 242 mL/g VS in Ze-MW, which was 9.1% higher than MW. Estimation of energy and assessment of cost revealed that the Ze-MW disintegration was profitable with a net yield of about 1986.8 INR /Ton of SS of biomass.

Thirdly, the impact of calcium specific chelant - Ethylene Glycol Tetra Acetic acid (EGTA) on EPS dissociation followed by biomass disintegration using Microwave (MW) was investigated. In the first phase of

the method, the EGTA dosage of 0.012 g/g SS was found to be optimal for dissociating the EPS. Subsequent disintegration of biomass in microwave (EGTA-MW) yielded a biomass liquefaction and solids reduction of about 39.7% and 30.5%. EGTA-MW disintegration reduces the amount of specific energy required to disintegrate the biomass from 18,900 kJ/kg TS to 13,500 kJ/kg TS, when compared to MW. The impact of EGTA-MW disintegration on anaerobic digestion was also evident from its methane yield (235.3 mL/g VS) which was 8.7% higher than MW. An economic assessment of this method provides a net profit of 638.4 INR / Ton of SS in EGTA-MW and highly endorsed for biomass disintegration.

Finally the efficiency of EPS dissociation was compared with combined disintegration. Combining H_2O_2 with MW resulted in a significant increment in biomass lysates liquefaction from 30% to 50% at 18,600 kJ/kg TS. The pH of H_2O_2 assisted MW disintegrated biomass (MW + H_2O_2) was in the alkaline range (pH: 9-10), and it made the biomass unfavourable for subsequent anaerobic digestion and inhibits methane production. In order to nullify the alkaline effect caused by the MW+ H_2O_2 combination, the addition of acid was considered for pH adjustment. Biomass liquefaction and solids reduction of about 56.1% and 33.1% was achieved in H_2O_2 assisted MW disintegrated biomass in acidic conditions (MW + H_2O_2 + acid) (COM-MW). COM-MW disintegration shows maximum methane production of 323 mL/g VS which was 12.8 % higher than MW. A cost analysis of this method reveals that COM-MW has the net profit of 1159.7 INR / Ton of SS of biomass.

A comparative analysis reveals that, even though the combined treatment yield higher liquefaction and biomethane production, in the economic point of view the biomass dissociated with H_2O_2 followed by the MW disintegration shows higher net profit than others.