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Research paper

Bio-methane potential test (BMP) using inert gas sampling bags with macroalgae feedstock

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ABSTRACT

An approach to Bio-methane potential test (BMP) was carried out at mesophilic temperature of 35 °C with Supel™ inert gas sampling bags as biogas collection and storage bags, using selected seaweed (macroalgae) as substrate. Samples were given a range of pre-treatments from washing, drying and macerating. Dried laminaria digitata (DD) with 68.14% VS (%TS) produced the highest BMP of 141 ± 5.77 L CH_4/kg VS, with methane content increasing to about 70%, while the lowest BMP of 93.35 ± 5.03 L CH_4/kg VS with methane content of about 65% was obtained for fresh laminaria digitata (FD) with 72.03% VS (% TS). Methane yields of 97.66 and 67.24 m^3 CH_4/t wet weight based on BMP results were obtained for DD and FD. Both DD and FD achieved within 28% and 38% of the theoretical BMP value based on the Buswell equation, respectively. The total methane *(V)* produced was computed based on;

$V = X_1 + X_2 - X_3$ corrected to Standard temperature and pressure (STP).

where X_1 = daily calculated headspace methane volume, X_2 = daily measured volume of methane in gas bags, X_3 = previous day headspace methane volume. An advantage of this approach is the volumetric measurement of gas produced directly from the gas bags, hence it does not require liquid displacement or pressure transducers. Results from a second set of freshly collected sample seaweed sample showed it was in agreement with published BMP values. All analysis were carried out without mineral supplementation.

1. Introduction

Anaerobic biodegradability (AB) is a terminology now used to describe Bio-chemical methane potential (BMP) [1–3]. It is defined as the fraction of compound(s) converted to biogas (methane and carbon dioxide) under oxygen-free conditions mediated by a diverse mixture of microorganisms for an indefinite degradation time. But in practice the degradation time is definite and methane potential estimated from extrapolation of the experimented degradation curve [4]. AB can be determined by the volume of biogas produced, or the amount of substrate depleted or the formation of intermediates and end products [3]. The biochemical methane potential (BMP) test is the procedure developed to measure the volume of methane produced [1,5]. The assay was developed as a standardized method to determine the ultimate biodegradability [6] and associated methane yield during the anaerobic methanogenic fermentation of organic substrates [7]. It is a proven and reliable method to obtain the extent and rate of organic matter conversion to methane [8]. The parameter, ultimate methane potential (λ_{max}) from the BMP assay is regarded to a great extent as the determining factor for both design and economic details of a biogas plant [5]. The experimental BMP approach is simple; a characterized [9] and quantified organic substrate is mixed with a known anaerobic inoculum in a suitable medium (minerals and water) under defined operating conditions where the gas evolved is quantified by a specified measurement system until gas production virtually ceases [10]. Mixtures of nitrogen (N_2) 70–80% and carbon dioxide (CO_2) 20–30% are used as headspace gas to create anaerobic conditions, these prevent pH - change in the water phase due to CO_2 from the headspace of the reactors [11], pure N_2 alone has been also used [10]. Blank controls are included to account for the biogas produced from the inoculum alone, these are termed endogenous tests [1]. The blank control gives an idea of the volume of biogas produced by the substrate alone [5]. Glass bottles with rubber septums as closed vessels are normally used (Fig. 1). The volume of the bottles range between 0.1 L and 2 L [5] to

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0.1–120 L [1], all depending on the homogeneity of the substrate used. It is recommended that samples and blank assay should be carried out in triplicate for statistical significance [5] because the BMP assay uses inoculum from different sources with varying quality and these can be relatively heterogeneous [10,11]. Furthermore, the biological approach in determining methane potential leads to substantial uncertainty hence triplicate samples should be used as a minimum [11].

Generally, the anaerobic biodegradability assay is used in triplicate [4]; to establish biodegradability of substrate for products (biogas/intermediates) formation, determination of the ultimate biogas potential and rate of biodegradation. In the first category, most methods are based on monitoring biogas using gasometric techniques [1,3–5,11] while different chemical analysis techniques are used to quantify formation of intermediates or substrate depletion [3]. In the gasometric methods, biogas is quantified either manometrically, by measuring pressure increase in constant volume or volumetrically as volume increase under constant pressure [1,3,5], and also by gas chromatography [1,2].

Volumetric methods comprise three approaches; displacement of a piston of a glass syringe inserted into the reactor, liquid displacement method using an alkaline solution for washing the biogas, or absorbing CO_2 and collection of the biogas in a gas sampling bag with low permeability [1], e.g. aluminium foil bags [12]. During the manometric method, biogas produced in the reactors creates a proportional overpressure which are measured by pressure transducers of various kinds [3]. Both methods require a complementary gas analyser to obtain percentage composition of methane in the biogas.

Seaweeds are marine macro-algae which can be biologically degraded to methane [13]. They can be utilized as a new promising biomass for the low-carbon economy, and recently have attracted attention as possible feedstocks for biorefinery ventures ([14]. Biorefineries are regarded as a sustainable technology that converts biomass into various marketable products, and energy [15]. Macroalgae have the potential of becoming a viable aquatic energy crop [16–18], but energy production from macroalgae is still limited due to economic viability [19]. Fig. 2 illustrates the current biofuel products from algae [19].

2. Materials and methods

2.1. Collection, pretreatment and storage

Algal biomass *Laminaria digitata (LD)* and *Laminaria Hyperborea*

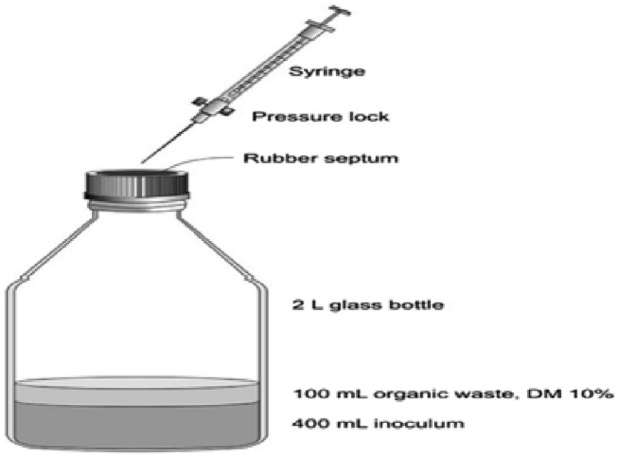

Fig. 1. Bio-methane potential reactor and sampling illustration [11].

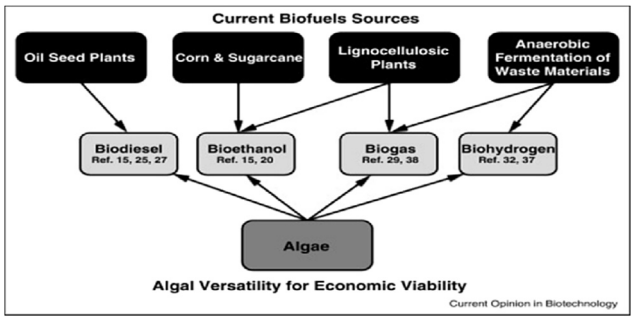

Fig. 2. Current renewable fuel sources from algae [17].

(LHY) used in the batch experiments were freshly collected from shallow water during low tide at Culler coats Bay, Tyne and Wear (NZ3572) on 19th December, 2013. The seaweed were transported in 1 m bags and were immediately washed to remove marine salts and sediments which can cause mechanical problems in digesters. Sand is known to be abrasive to moving parts such as mixers and pumps while salt removal leads to more stable digestion [20].

In preparation of the feedstocks, only the frond was used for *LD,* while stipe (stem) were used for the *LHY.* Two categories of pretreatment were carried out on both samples to obtain fresh slurry and a dried algal powder. For the slurry the fronds were roughly chopped by hand to particle size of about 10 mm, while the stipe was broken to smaller pieces <5 mm using knives and hammer mill. Approximately 250 g of each were then macerated with 250 ml of distilled water using a kitchen blender to give consistent thick slurry (particles generally <2 mm) suitable for direct addition to the reactors. The algal powder was obtained by oven dying the sample at 104 ^{O}C for 24 h and then pulverized with a Kenwood 100 coffee blender to particle size generally <1 mm. Both types of pretreated sample were labelled in 1 L containers and stored at 4 °C until required.

2.2. Inoculum

The specific methanogenic activity test (SMA) is normally used to check the quality of inoculum in anaerobic digesters. It is an indication of the efficiency of anaerobic treatment process because it measures the rate of the methanogenic activity under defined substrate conditions [21]. The SMA test is a quick and simple way to get information about the percentage of active methanogenic microorganism in a sludge, and also estimate the rate of maximum methane production of a reactor at a particular sludge density [22], or capability [23] to convert volatile fatty acids into methane under ideal conditions [23]. The test is performed with acetate, or acetic acid, or mixture of acetic, propionic and butyric acids [24], because in non-gastrointestinal environments like anaerobic digesters, acetate is one of the major intermediates of fermentation [22] and is regarded as the principle precursor of about 70% of methane produced under typical operating conditions [25]. The inoculum used was collected from laboratory scale mesophilic anaerobic digesters running in the environmental engineering laboratory, Newcastle University. It had been stored at 4 °C for between 1 and 4 weeks before use, and had the following characteristics; pH 7.33, 13.95% TS and 58.77% VS (%TS). The inoculum was pre-incubated using 2 L reactor bottles at 35 °C for 3 days with waste beer COD concentration 117 g/L to restore/reactivate the methanogenic activity. Active biomass was confirmed by good biogas production (1 L biogas/L reactor/d) with 50–70% methane content in the biogas (Fig. 3).

Before using the pre-incubated inoculum for both SMA and BMP tests it was de-gassed between 3 and 5 days until biogas production